# TREATMENT OF PETROLEUM REFINERY WASTEWATER IN AN INNOVATIVE SEQUENCING BATCH REACTOR

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# Declaration

The research reported in this thesis was conducted at Liverpool John Moores University, Department of Civil Engineering, between September 2014 and June 2018. I declare that the work is my own and has not been submitted for a degree at another university.

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# Abstract

The difficulty with sludge settleability is considered one of the main drawbacks of sequencing batch reactors. The aim of this study therefore is to improve sludge settleability by introducing a novel, two-stage settling sequencing batch reactor (TSSBR) separated by an anoxic stage. The performance of the TSSBR was compared with that of a normal operating sequencing batch reactor (NOSBR), operating with the same cycle time.

The results show a significant improvement in sludge settleability and nitrogen compound removal rates for the TSSBR over the NOSBR. The average removal efficiencies of ammonia-nitrogen ($NH_3$-N), nitrate-nitrogen ($NO_3$-N) and nitritr-nitrogen ($NO_2$-N) have been improved from 76.6%, 86.4% and 87.3% respectively for the NOSBR to 89.2%, 95.2% and 96% respectively for the TSSBR. In addition, the average sludge volume index (SVI) for the NOSBR has been reduced from 42.04 ml/g to 31.17 ml/g for the TSSBR. After three months of operation, there was an overgrowth of filamentous bacteria inside the NOSBR reactor, while the morphological characteristics of the sludge inside the TSSBR reactor indicated a better and homogenous growth of filamentous bacteria.

TSSBR system proves to be more efficient than NOSBR by improving the sludge settleability and enhancing nitrogen compounds' removal efficiency, therefore, the TSSBR operating conditions including (mixed liquor suspended solids, hydraulic retention time, fill conditions, fill time, volumetric exchange rate, organic loading rate and hydraulic shock) have been optimised to obtain the optimal performance of the TSSBR system regarding the treatment efficiency and sludge settling performance.

The results of optimising the TSSBR operating conditions are as follows: the optimal MLSS range was 3000 mg/l to 4000 mg/l; the optimal HRT was 6 h; unaerated feeding was better than the aerated feeding, and 15 minutes was the optimal feeding time; the optimal VER value was 20%; the optimal OLR ranges were 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate.

Finally, the TSSBR system was operated under the obtained optimal operating conditions. The results showed that the treatment efficiency of COD and $NO_3$-N had been improved significantly. Although the removal efficiency of $NH_3$-N and $NO_2$-N did not improve, the removal efficiency of both is more than 90%, which is considered a good treatment efficiency for the TSSBR system. In addition, the settling performance of the TSSBR was significantly improved after operating the system under the optimal operating conditions.

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## List of Abbreviations

| | |
|---|---|
| AGS | Aerobic granular sludge |
| ASP | Activated sludge process |
| ASS | Activated sludge system |
| ASSBR | Aerobic suspension-sequencing batch reactor |
| BNR | Biological nutrient removal |
| BOD | Biochemical oxygen demand |
| BTEX | Benzene, toluene, ethylbenzene, xylenes |
| C/M | Carbon / microorganisms ratio |
| C:N:P | Carbon : Nitrogen : Phosphorus ratio |
| cBOD | Carbonaceous biochemical oxygen demand |
| COD | Chemical oxygen demand |
| DCS | Double-cycle shock |
| DO | Dissolved oxygen |
| Draw | Discharging the treated wastewater from the SBR tank |
| F/M | Food / microorganisms ratio |
| GAC | Granular activated carbon |
| GAC-SBR | Granular-activated carbon sequencing batch reactor |
| GSBR | Granular sequencing batch reactor |
| HRT | Hydraulic retention time |
| MLSS | Mixed liquor suspended solids |
| MLVSS | Mixed liquor volatile suspended solids |
| nBOD | Nitrogenous biochemical oxygen demand |
| NO | Normal operation |

| | |
|---|---|
| NOSBR | Normal operating sequencing batch reactor |
| OCO | Operating condition optimisation |
| OLR | Organic loading rate |
| ORP | Oxidation-reduction potential |
| PAC | Powdered activated carbon |
| PAHs | Polyaromatic hydrocarbons |
| PC | Personal computer |
| PRW | Petroleum refinery wastewater |
| RAS | Return activated-sludge |
| RC | Recovery condition |
| React | Reaction stage in the SBR |
| RPM | Revolutions per minute |
| SBR | Sequencing batch reactor |
| SCS | Single-cycle shock |
| SEM | Scanning electron microscopy |
| SR | Standardised residuals |
| SRT | Solid retention time |
| SSV | Settled sludge volume |
| SVI | Sludge volume index |
| Tf | Filling time |
| TL/MLSS | Total filament length per MLSS |
| TL/Vol | Total filament length per sample volume |
| TN | Total nitrogen |
| TOC | Total organic carbon |

| | |
|---|---|
| TSS | Total suspended solids |
| TSSBR | Two-stage settling sequencing batch reactor |
| UASB | Up-flow anaerobic sludge blanket |
| UK | United Kingdom |
| VER | Volumetric exchange rate |

# List of Nomenclature

| | |
|---|---|
| B | Boron |
| C | Carbon |
| C° | Celsius degree |
| $C_{10}H_{19}O_3N$ | Biodegradable organic matter in wastewaters |
| $C_6H_{12}O_6$ | Glucose |
| Ca | Calcium |
| $CaC_{l2}.2H_2O$ | Calcium chloride dihydrate |
| $CH_3COOH$ | Acetic acid |
| $CH_3OH$ | Methanal |
| $CH_4$ | Methane |
| CI | Chlorine |
| CO | Carbon monoxide |
| $CO_2$ | Carbon dioxide |
| Cr | Chromium |
| Cu | Copper |
| F | Fluorine |
| Fe | Iron |
| $FeCl_3.6H_2O$ | Iron(III) chloride hexahydrate |
| H | Hydrogen |
| $H_2O$ | Water |
| $HCO_3$ | Bicarbonate |
| K | Potassium |
| $KH_2PO_4$ | Monobasic potassium phosphate |

| | |
|---|---|
| $KNO_3$ | Potassium nitrate |
| Mg | Magnesium |
| $MgSO_4.7H_2O$ | Magnesium sulphate heptahydrate |
| Mn | Manganese |
| Mo | Molybdenum |
| N | Nitrogen |
| $N_2$ | Nitrogen gas |
| $N_2O$ | Nitrous oxide |
| Na | Sodium |
| $NaHCO_3$ | Sodium bicarbonate |
| $NH_3$-N | Ammonia-nitrogrn |
| $NH_4$ | Ammonium |
| $NH_4Cl$ | Ammonium chloride |
| Ni | Nickel |
| NO | Nitric oxide |
| $NO_2$ | Nitrite |
| $NO_2$-N | Nitrite-nitrogen |
| $NO_3$ | Nitrate |
| $NO_3$-N | Nitrate-nitrogen |
| $O_2$ | Oxygen |
| OH | Hydroxide |
| P | Phosphorus |
| pH | Measure of the acidity or basicity of an aqueous solution |
| S | Sulfur |

| | |
|---|---|
| Se | Selenium |
| Si | Silicon |
| Zn | Zinc |
| $\theta_c$ | Mean cell residence time |

# Glossary

| | |
|---|---|
| Autotrophic bacteria | Autotrophic bacteria are those that have inorganic carbon sources (primary producers), which they use to produce their own organic nourishment |
| Endogenous decay | Refer to the cell mass due to oxidation of internal storage products for energy for cell maintenance, cell death |
| Exogenous source | External source such as methanol in denitrification process |
| Heterotrophic bacteria | Heterotrophic bacteria use organic carbon sources, or in other words, other living organisms or derivatives from them to produce their own organic nourishment |
| Settle | Settling stage in the SBR |

# CHAPTER 1

# Introduction

## 1.1 Background

### 1.1.1 General introduction

The petroleum refinery industry produces more than 2,500 refined products from crude oil; these products include gasoline, liquefied petroleum gas, aviation fuel, kerosene, fuel oils and diesel fuel (Benyahia et al., 2006).

A considerable amount of water is used in the refinery processes, mainly for cooling, distillation, hydrotreating and desalting systems (Benyahia et al., 2006). The amount of refinery wastewater generated and its characteristics depend on the process design. The Refinery industry discharges a huge amount of polluted wastewater, containing phenol levels of 20–2000 mg/l; COD levels of approximately 300–600 mg/l; benzene levels of 1–100 mg/l; 0.1–100 mg/l for chrome and 0.2–10 mg/l for lead; and other trace elements (World Bank Group, 1999).

In addition, petroleum refinery wastewater may contain aliphatic and aromatic petroleum hydrocarbons, which may affect negatively on the surface of the soil and aquatic life (Sun et al., 2008)

### 1.1.2 Environmental effects of petroleum refinery wastewater

During oil and gas exploration and production operations at oil fields, a huge amount of polluted water containing petroleum hydrocarbons is produced (Ghorbanian et al., 2014; Tong et al., 2013). Untreated water discharges may be toxic to the environment due to its characteristics of hydrocarbons, dissolved solids, and trace elements. It contains different types of hydrocarbons with different structural and chemical properties (Tellez et al., 2002). Therefore, petroleum refinery wastewater (PRW) threatens environmental health due to its high toxicity (Bakke et al., 2013).

### 1.1.3 Biological wastewater treatment

Wastewater treatment plants are designed to remove organic and inorganic aqueous pollutants that affect negatively on human health and water bodies. In order to protect the water bodies that directly receive effluent from wastewater treatment plants, environmental agencies regulate limits for a range of substances classified as toxic or dangerous.

There are a significant number of technologies available for the treatment of industrial wastewater; biological treatment is no exception. The latter is considered one of the most convenient technologies for the treatment of industrial wastewater due to its manufacturing and operational cost requirements. In addition to cost considerations, biological treatment has proved to be an effective technology for removing high concentrations of pollutants.

One of the common biological technologies is the activated sludge process (ASP), used worldwide for the treatment of domestic and industrial wastewater (Jassby et al., 2014). It consists of several reactors in which microorganisms degrade incoming wastewater and in doing so, grow and produce new microorganisms. After degradation is achieved, these microorganisms are separated from the treated wastewater by sedimentation. In order to sustain an active and high concentration of solids for the reaction treatment, some sediment solids should be removed from the system, others recycled back into the aeration basin (Jones and Schuler, 2010). One of the drawbacks of ASP is that it requires a large footprint for its treatment tanks (Chen et al., 2013).

Often industries are located in cities, which makes it difficult to build a treatment system containing several tanks. In this case, alternatives are available such as sequencing batch reactors (SBR).

### 1.1.4 Sequencing batch reactor

SBR is one of the alternatives of the activated sludge process that work on the same principles which is biological wastewater treatment technology. It has been treating successfully both municipal and industrial wastewater (Bagheri et al., 2015).

In addition, SBR is a fill and draw type sludge system that has five basic operating modes - Fill, React, Settle, Draw and Idle (Environmental Protection Agency, 1999), which operates in time instead of space. In one tank, SBR performs equalization, neutralization, biological treatments and secondary sedimentation via timed control sequence (Alattabi et al., 2015). Over the recent years, SBR technology has become an attractive technology due to its unique design and ease of industrialisation. The difference that made SBR overcome the conventional activated sludge system is that the latter requires many tanks to operate while the SBR system could be operated in a single tank. The SBR system consists of the following basic steps (Mata et al., 2015; Sutton and Mishra, 1990):

1. Fill: In the fill stage, the wastewater and substrate are added for microbial activity. It can be static fill, mixed fill, or react fill. In the static fill, the wastewater influent is introduced to the system without mixing or aeration. Mixed fill involves turning on a mixing device during the fill phase. While aeration is turned on during the fill phase in the react fill mode of operation.

2. React: The objective of this stage is to let the bacteria biodegrade the coming organic matter and other pollutants. It could consist of mixing or aeration, or a combination of both.

3. Settle: During the settle phase, liquid-solid separation occurs.

4. Draw: In this stage, the effluent is decanted from the reactor.

5. Idle: It is the final stage in an SBR system and is only used in multi basin applications.

Sludge waste will be achieved during the idle phase. Figure 1.1 shows a typical schematic diagram of SBR process.

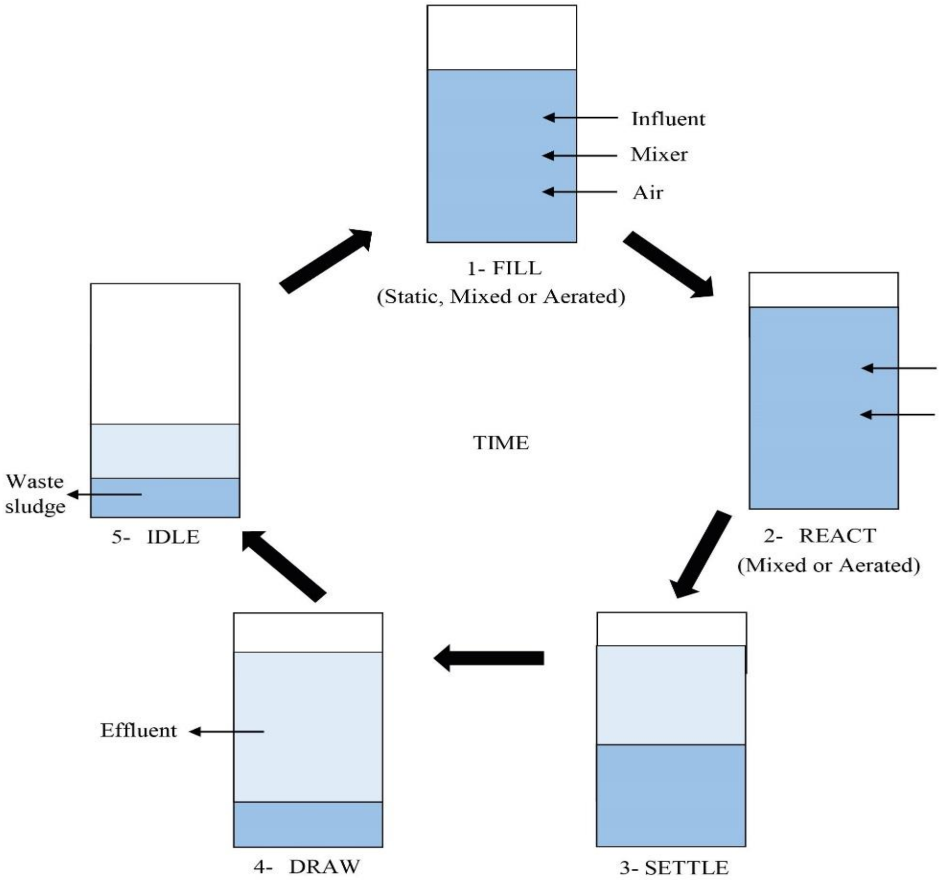



Figure 1.1: Sequencing batch reactor

Source (Wilderer et al., 2001)

## 1.2 Problem statement

SBR is an activated sludge process that consists of a sequence of stages which operates in one tank in a time sequence; these stages are: fill, react, settle, draw and idle. It has been reported that SBR requires less area, is flexible to operate and could be operated automatically (Miao et al., 2014; Abu Hasan et al., 2016). However, solid-liquid separation or sludge bulking is still one of the most problematic issues with SBR and ASP in general (Chen et al., 2013; Guo et al., 2014a; Guo et al., 2014b; Iritani et al., 2015; Jin et al., 2003; Koivuranta et al., 2013; Koivuranta et al., 2015; Mesquita et al., 2011; Tansel, 2018; Wilen et al., 2008; Xia et al., 2016; Yang et al., 2017; Ye et al., 2016; Zhang et al., 2017)

Researchers have been reporting several reasons related to this problem such as difficulty of handling sudden changes in the operating parameters (Mesquita et al., 2011), microbial clustering behaviour (Ye et al., 2016), the overgrowth of filamentous bacteria (Eikelboom, 2000; Guo et al., 2012; Jenkins et al., 2003; Martins et al., 2004; Mesquita et al., 2011), foaming (Guo et al., 2014a; Guo et al., 2012), pin-point sludge (Guo et al., 2012; Jenkins et al., 2003), poor macrostructure (Guo et al., 2012), poor flocculation properties (Contreras et al., 2004; Jenne et al., 2007; Jin et al., 2003), floc size distribution (Amaral and Ferreira, 2005; Grijspeerdt and Verstraete, 1997; Jin et al., 2003; Mesquita et al., 2011; Schmid et al., 2003).

To overcome the settling problem in the SBR technology, researchers have been trying different solutions, one of them is granulation technology. In a specific environment, microbial self-agglomeration forms a granular biological polymer which is known as aerobic granular sludge (AGS) (Kreuk et al., 2007; Long et al., 2016). It has many advantages such as high degradation ability, significant settling velocity, regular shape and compact structure (Adav et al., 2008a; Chen et al., 2013; Long et al., 2016; Show et al., 2012; Zhang et al., 2015). However, AGS stability might decline after a long period of operation (Adav et al., 2008b; Lee et al., 2010; Liu and Liu, 2006; Liu et al., 2004; Tay et al., 2002). In addition to the stability loss, granulation technology has other problems such as producing high operation temperature, needing a long acclimatisation time and not being efficient with a low concentration of organic wastewater (Lettinga et al., 1980; Qin et al., 2004), which makes granulation technology need more research to tackle these issues.

Another attempt to overcome the settling problem is chemical addition before the settling stage to improve the settling performance (Agridiotis et al., 2007; Wu et al., 1997). However, this procedure could raise the cost of treatment and results in more complex and toxic residual which affect negatively on the environment (Iritani et al., 2015).

Along with granulation sludge technology and chemical conditioning, researchers have been modifying the operation strategy or adding more stages to the SBR treatment cycle as a trial to improve the treatment performance without additional cost if the cycle time did not increase (Aziz et al., 2011; Chen et al., 2013; Mata et al., 2015). The inspiration for this research is grounded in the above modifications and trials, to introduce a novel, two-stage settling SBR.

This system will focus on three issues. The first would be to create a shock after the first settling stage and allow small flocs to cling together, merge with large flocs and settle again in the second settling stage. Secondly, examination of the effect of this procedure, the elimination of filamentous accumulation and improvement in the settling stage. Finally, verification of whether separating the two stages of settling with an anoxic stage enhances nitrogen removal efficiency by improving the denitrification stage.

## 1.3 Aims and Objectives

The aims of this research project are:

1- To improve the settling phase and minimise the operating power by developing an innovative design for the SBR optimising the process variables to result in a more robust and efficient process. The introduction of a two stage-settling phase sequence in the SBR system instead of one settling phase will be developed by running a short period of mixing between them to enhance the flocculation and improve settling, as well as improving the nitrogen removal efficiency.

2- To optimise the TSSBR design operating conditions (mixed liquor suspended solids, hydraulic retention time, fill conditions, fill time, volumetric exchange rate, organic loading rate and hydraulic shock) to find the optimal performance of the TSSBR system.

Objectives:

1- Conduct a critical literature review to make an experiment design choice.

2- To determine the removal percentages of chemical oxygen demand (COD), ammonia nitrogen, nitrate-nitrogen and nitrite-nitrogen from synthetic PRW for both NOSBR and TSSBR

3- To study the mixed liquor suspended solids in different concentrations and their impact on the treatment efficiency and sludge settleability in the TSSBR system.

4- To examine the fill conditions and find their impact on the treatment efficiency and sludge settleability in the TSSBR system.

5- To find the effect of VER on the treatment efficiency and sludge settleability in the TSSBR system.

6- To determine the effects of hydraulic retention time on the treatment efficiency and sludge settleability in the TSSBR system by studying different HRTs (4, 6, 8 and 12 hrs).

7- To study the effects of organic loading rate on the treatment efficiency and sludge settleability of the TSSBR by gradually increasing the concentration of glucose and potassium-nitrate.

8- To examine the capability of the TSSBR of handling hydraulic shock by decreasing the cycle time suddenly.

9- To study the dissolved oxygen (DO), pH, temperature and oxidation-reduction potential (ORP) of the SBR system and relate this to biological treatment of petroleum compounds.

## 1.4 Originality of the research:

1- The settling stage is an important phase in the SBR system and it is a time-controlled cycle. However, many researchers have reported poor, slow or incomplete particle settling in the settle phase of the SBR system. This research project innovates two-stage settling SBR system instead of one settle stage by running a short period of mixing between the two phases to enhance the flocculation and improve settling. In addition, nitrogen removal efficiency could be enhanced in this innovative cycle of SBR.

2- Mixed liquor suspended solids (MLSS) is an important factor affecting the efficiency of the SBR system. Many researchers such as (Elmolla et al., 2012; Martins et al., 2003; Tsang et al., 2007) have studied the effects of MLSS on the SBR system. However, the relationship between MLSS and sludge settleability has not been studied. This research project will investigate the effects of different concentrations of mixed liquor suspended solids (MLSS) (±2000, and ±3000, ±4000 and ±6000 mg/l) on sludge settleability and their impact on effluent quality by studying the sludge characteristics and treatment efficiency for each run in the TSSBR.

3- The effects of fill conditions on sludge settleability have not been considered (Miao et al., 2015; Moussavi et al., 2010; Thakur et al., 2013b). In this research project, the effects of aerated and un-aerated fill as well as fill time on sludge settleability in the TSSBR system will be investigated.

4- Many researchers such as (Leong et al., 2011; Rodriguez-Caballero et al., 2015; Thakur et al., 2013b; Thakur et al., 2014) have studied the effects of HRT on the SBR system because HRT is considered one of the most significant parameters in the SBR system. However, the relationship between HRT and sludge settleability has not been studied. In this research project, the relationship between HRT and sludge settleability in the TSSBR system will be explored.

## 1.5 Scope of work

This research project was performed to treat and improve the quality and enhance the settleability of synthetic wastewater using a two-stage settling sequencing batch reactor. The performance of the two-stage settling sequencing batch reactor was compared with that of a normal operating sequencing batch reactor, operating with the same cycle time

The synthetic wastewater used in this research contains eight chemical compositions, which are glucose, magnesium (II) sulphate heptahydrate, sodium bicarbonate, monobasic potassium phosphate, calcium chloride dehydrate, iron (III) chloride hexahydrate, potassium nitrate and ammonium chloride. The chemical compositions were added to the treatment reactor with different concentrations. The capacity of the treatment reactor was 5 L and the treatment process had been achieved through accumulated biomass. The goal was to remove the undesirable chemicals such as COD, ammonia-nitrogen, nitrite-nitrogen and nitrate-nitrogen from the influent wastewater and examine the solids' settleability in different treatment conditions for both systems and compare the results to find the efficiency of the two-stage settling sequencing batch reactor.

Then the operating conditions of the two-stage settling sequencing batch reactor have been optimised to find the optimal performance of the TSSBR system. The online monitoring of pH, DO and ORP profiles were recorded from both systems and correlated with removal rates of COD, ammonia-nitrogen, nitrite-nitrogen and nitrate-nitrogen in the process.

Finally, the TSSBR system was operated under the optimal operating conditions to achieve the optimal performance of the TSSBR system.

## 1.6 Thesis outline

Chapter 1 illustrates the solid settling problems in the SBR and what is the possible solution to treat them. In addition, the main aims, objectives, research novelty and scope of the study were illustrated briefly. Chapter 2 includes the characteristics of petroleum refinery wastewater and its treatment technologies; the most theoretical and general works associated to SBR and the current solutions for its settleability problems in addition to the online monitoring of the parameters of pH, DO and ORP. Chapter 3 describes the design of the innovative two stage-settling SBR and the methodology used in this research which contains the materials and methods of measuring the parameters and sampling. Also, it contains the instruments and devices used in this study. In addition, the morphological study and the image analysis procedure are described. Chapter 4 shows the results and discussion of the treatment efficiencies and solids settling performance of the two-stage SBR system and compares it with the results of the normal operating SBR system. Chapter 5 illustrates the TSSBR operating conditions optimisation and the TSSBR performance under the optimal conditions. Chapter 6 concludes the overall results of the current study and recommendations for future studies.

# CHAPTER 2

# Literature review

The literature review will consist of five parts: the first part will be focused on the PRW and its characterisation and treatment methods. The second part will discuss biological treatment technology used to treat PRW. The third part will focus on the SBR system and its operating conditions which have been customised by many researchers to approach a maximum removal of undesired wastewater components. The fourth part will talk about the solids settleability problems and their solutions. The last part will discuss the online monitoring for nutrient removal.

## 2.1 Petroleum refinery wastewater

Wastewater treatment performs natural purification processes to the maximum level possible. It is also designed to implement these processes in a planned environment (Spellman, 2003). In addition, another goal for the treatment plant is to treat the nutrients that are not commonly conducted to natural processes, and also remove the solids generated within the treatment unit steps. Wastewater treatment plant is designed to carry out different goals: preserve (public health, public water supplies and aquatic life), maintain the superior uses of the waters and preserve close lands. Figure 2.1 illustrates the sequence steps in wastewater treatment. Each step can be adapted using one or more treatment mechanisms (Spellman, 2003).

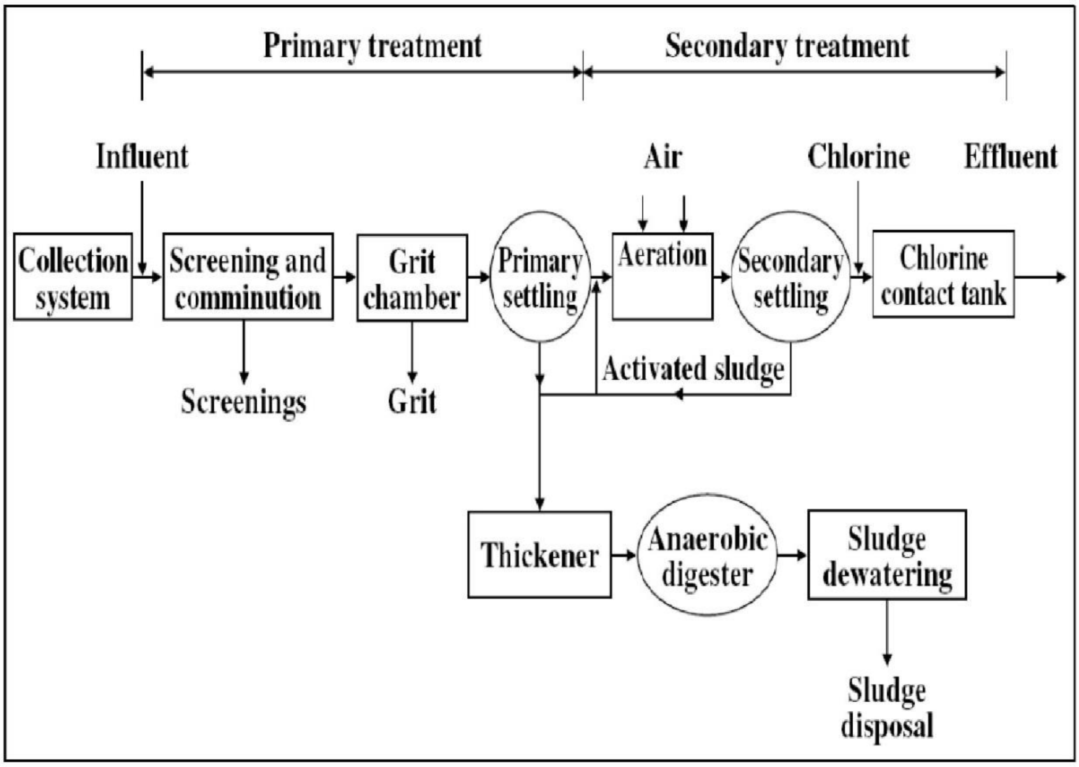



Figure 2.1: Wastewater treatment flow chart
(Metcalf and Eddy, 2014)

Petroleum refinery wastewater (PRW) is wastewater discharging from industries related to manufacturing fuels and refining crude oil (Harry, 1995). PRW contains oil and grease along with other trace nutrients (Wake, 2005). The generation of PRW is a critical matter globally as a consequence of rising energy demands, which will increase the processing of crude oil (Doggett and Rascoe, 2009).

### 2.1.1 Classification of petroleum refinery wastewater

PRW contains organic and inorganic compounds, suspended solids, water-soluble metals, dissolved formation minerals, dispersed and dissolved oil, polyaromatic hydrocarbons (PAHs), hydrocarbons such as BTEX and phenol (Khaing et al., 2010; Ma and Guo, 2009; Razi et al., 2009). To treat these pollutants, crude oil requires a desalting process using huge amounts of water (Diya'uddeen et al., 2015).

Table 2-1 shows the composition of PRW, which depends on the complexity of the refining process.

Table 2-1: Characteristics of petroleum refinery wastewater

| Parameter | (Dold, 1989) | (Coelho et al., 2006) | (Mizzouri and Shaaban, 2013) | (Ma and Guo, 2009) | (Ahmed et al., 2011) | (Khaing et al., 2010) | (Thakur et al., 2013a) |
|---|---|---|---|---|---|---|---|
| $BOD_5$, mg/l | 150-350 | 570 | 240 | 150-350 | - | - | - |
| COD, mg/l | 300-800 | 850-1020 | 920 | 300-600 | 1066 | 330-556 | 350±25 |
| Phenol, mg/l | 20-200 | 98-128 | 12.6 | - | - | - | 10 |
| Oil, mg/l | 3000 | 12.7 | 210 | 50 | - | 40-91 | - |
| Total suspended solids, mg/l | 100 | - | 122 | 150 | 189.9 | 130-250 | - |
| Sulphate, mg/l | - | - | - | - | 22.6 | - | 120 |
| Nitrate, mg/l | - | - | - | - | 0.47 | - | 3.7 |
| BTEX, mg/l | 1-100 | 23.9 | - | - | - | - | - |
| Ammonia, mg/l | - | 5.1-2.1 | 23.4 | 10-30 | 7.8 | 4.1-33.4 | - |
| pH | 8.0-8.2 | 8.0-8.2 | 8.9 | 7-9 | 6 | 7.5-10.3 | 8±.5 |
| Turbidity, NTU | 22-52 | 22-52 | - | - | - | 10.5-159.4 | - |

### 2.1.2 Petroleum refinery wastewater treatment methods

The traditional treatment methods of refinery wastewater are the physical, chemical and biological treatment (El-Naas et al., 2014).

#### 2.1.2.1 Physical treatment

It is a wastewater treatment process in which the pollutants removed from the wastewater by physical forces. Examples of the physical treatment methods are screening, sedimentation, flocculation, mixing, filtration, flotation and adsorption (Metcalf and Eddy, 2014).

#### 2.1.2.2 **Chemical treatment**

In this type of wastewater treatment, the removal of pollutants is done by chemical addition or chemical reactions. Gas transfer, precipitation and adsorption are examples of the chemical treatment techniques. Precipitation is a common chemical treatment unit, in which a chemical precipitate is produced, and then it can be removed through a membrane process, filtration or settling. Gas transfer is another chemical treatment method; a common example of this process is aeration, in which the oxygen is added to the water to support the aerobic reaction. Another common chemical unit process is the use of chlorine for wastewater disinfection, which has been practised for more than a century (Metcalf and Eddy, 2014).

#### 2.1.2.3 **Biological treatment**

In this type of treatment, the pollutants are removed by bacterial activity or other microorganisms. It is used to remove dissolved or colloidal organic substances. Biological treatment work by converting these substances into (1) biological cell tissue which can be settled in the clarifier and (2) gases which then will be released into the atmosphere. Nitrogen and phosphorous can be removed by biological treatment. In addition, biological treatment could treat most types of wastewater if a proper control of the treatment environment is provided. Thus, it is the responsibility of the wastewater engineer to ensure that the appropriate environment is produced and controlled effectively to achieve all treatment objectives (Metcalf and Eddy, 2014).

### 2.1.3 UK wastewater discharge regulations

The Environment Agency regulates wastewater treatment works (WWTW) by assessing the quality of the wastewater they discharge against set compliance limits as shown in Table 2-2 (Environment Agency, 2018).

Table 2-2: UK wastewater discharge regulations

| Parameter | Concentration | Minimum removal percentage |
|---|---|---|
| BOD | 25 mg/l | 70-90 |
| COD | 125 mg/l | 75 |
| Total phosphorus | 2 mg/l | 80 |
| Total nitrogen | 15 mg/l | 70-80 |

## 2.2 Biological treatment for petroleum refinery wastewater

Biological processes use bacteria or other types of microorganisms to biodegrade the organic matter into simple products ($CO_2$, $H_2O$ and $CH_4$) under aerobic, anaerobic or semi-aerobic conditions (Razi et al., 2009; Ma and Guo, 2009). A carbon: nitrogen: phosphorus (C:N:P) ratio (100:5:1) is adequate for microorganisms to grow (Chan et al., 2010; Metcalf and Eddy, 2014). A study on biodegradation of petroleum oil by nematodes has identified Bacillus sp. as a primary degrader and cooperation with nematodes for degradation of pollutants (Chan, 2011). In a study using bioaugmentation, activated sludge system (ASS) took only 20 days to achieve COD below 80 mg/l (84.2% COD removal efficiency) and $NH_4$-N concentration of 10 mg/l compared to the non-bioaugmentation system, which needed an extra 10 days to reach similar effluent quality (Ma and Guo, 2009) . The biological process is classified as suspended-growth, attached-growth or hybrid process.

### 2.2.1 Suspended growth

In this process, bacteria are kept in a system of a batch reactor in suspension mode within the liquid. The batch reactor allows operating with mixing under aerobic or anaerobic environment. One of the common suspended-growth processes is activated sludge process. Common examples of activated sludge process are complete mix, plug-flow and sequencing batch reactor. While plug-flow and complete mix activated sludge require return activated-sludge (RAS) system and clarifier, SBR operates without a clarifier (Metcalf and Eddy, 2014).

### 2.2.2 Attached growth

In this process, bacteria are attached to a medium (rocks, slag or plastic), which enables them to create biofilm containing extracellular polymeric substances produced by the bacteria (Hsien and Lin, 2005; Metcalf and Eddy, 2014). Bioreactors with adhered biofilm have a greater concentration of biomass retained in the system with greater metabolic activities (Muneron de Mello et al., 2000).

### 2.2.3 Hybrid system

This process is a combination of attached-growth and suspended growth process in the same reactor as the combination of activated sludge and submerged biofilters (fixed bed biofilters). A carrier material in the reactor is maintained in suspension by aeration or mechanical mixing (moving bed reactor) (Metcalf and Eddy, 2014).

### 2.2.4 Microbial growth

Different kinds of microorganisms, mainly bacteria are the responsible for removing dissolved and particulate carbonaceous biochemical oxygen demand (cBOD) and biodegrading the organic matter by biological activity. The dissolved and particulate carbonaceous organic matter is oxidized by microorganisms to convert them into simple end products and additional biomass.

Bitton (2005) defines the growth of microbial as an increase in microbial mass. There are chemical and physical parameters affecting the growth of microbial:

1. Temperature is an important factor affecting the microbial growth. The growth can occur at temperatures below freezing or up to more than 100°C. Based on the appropriate growth temperature, microbial can be classified as thermophiles, psychrophiles and mesophiles. Psychrophiles can grow at low temperatures while thermophiles can grow at high temperatures.

2. pH: the suitable pH for microbial growth is around 7. The studies have shown that the biological treatment occurs basically at neutral pH. The growth of microbial results in a decline in the pH of the medium. Conversely, some microbial can increase the pH of their medium such as denitrifying bacteria.

3. Oxygen level: the microbial can grow in the presence or absence of oxygen. The microbial is divided into strict aerobes, strict anaerobes and facultative anaerobes which can grow in the absence or presence of oxygen. Some microbial are microaerophilic which can be grown best at low levels of oxygen.

4. Moisture: the microbial must have a supply of water available. The effect of low water levels slows down the growth, but the amount of water for growth varies between the species.

5. Nutrient content: water, carbon, nitrogen, vitamins, minerals and energy source are the requirements for microbial growth.

## 2.3 Biological BOD and COD removal

Biochemical oxygen demand (BOD) is a measure of the amount of oxygen that bacteria will consume while decomposing organic matter under aerobic conditions. Biochemical oxygen demand is determined by incubating a sealed sample of water for five days and measuring the loss of oxygen from the beginning to the end of the test. Samples often must be diluted prior to incubation or the bacteria will deplete all of the oxygen in the bottle before the test is complete (Metcalf and Eddy, 2014).

The main focus of wastewater treatment plants is to reduce the BOD in the effluent discharged to natural waters. Wastewater treatment plants are designed to function as bacteria farms, where bacteria are fed oxygen and organic waste. The excess bacteria grown in the system are removed as sludge, and this “solid” waste is then disposed of on land (Hami et al., 2007).

Chemical oxygen demand (COD) does not differentiate between biologically available and inert organic matter, and it is a measure of the total quantity of oxygen required to oxidize all organic material into carbon dioxide and water. COD values are always greater than BOD values, but COD measurements can be made in a few hours while BOD measurements take five days (Ramanand Bhat et al., 2003). BOD and COD can be removed biologically using different types of technologies such as SBR (Jena et al., 2016).

## 2.4 Biological nitrogen removal

Due to its contribution in the eutrophication, nitrogen has to be removed from wastewater before discharge to the water bodies. Nitrification and denitrification are the main two stages of removing the nitrogen biologically (Environmental Protection Agency, 1993).

### 2.4.1 Nitrification

Nitrification is the first stage in biological nitrogen removal, and it consists of two steps: converting the ammonia to nitrite ($NO_2$-N) and then converting the nitrite to nitrate ($NO_3$-N). These two steps of nitrification happen under an aerobic environment in which the oxygen plays the role of electron accepter. The nitrification is an essential process as it is responsible for removing the ammonia from the wastewater biologically and consequently preventing the fish toxicity and reducing the eutrophication (Metcalf and Eddy, 2014).

In the activated sludge process, nitrification is achieved by two distinctly different groups of aerobic autotrophic bacteria. Ammonia Oxidizing Bacteria (AOB) is the first group of autotrophic bacteria that are responsible for oxidising the ammonia to nitrite (Equation 2.1). Nitrite Oxidizer Bacteria (NOB) is the second group of autotrophic bacteria that are responsible for oxidising the nitrite to nitrate (Equation 2.2).

$$NH4^{+} + \frac{3}{2}\ O_2 \longrightarrow NO_2^{-} + H_2O + 2H^{+} \qquad (2.1)$$

$$NO^{-2} + \frac{1}{2}\ O_2 \longrightarrow NO_3^{-} \qquad (2.2)$$

Thus, (Equation 2.3) shows the total oxidation of ammonia to nitrate:

$$NH4^{+} + 2O_2 \longrightarrow NO_3^{-} + 2H^{+} + H_2O \tag{2.3}$$

These two groups of aerobic autotrophic bacteria acquire energy for surviving from inorganic nitrogen compounds' oxidation, by using inorganic carbon as a source of their required cellular carbon. In addition, the amount of alkalinity needed to complete the reaction (Equation 2.3) can be calculated according to the Equation 2.4:

$$NH4^{+} + 2HCO_3^{+} + 2O_2 \longrightarrow NO_3^{-} + 2CO_2^{+} + 3H_2O \tag{2.4}$$

### 2.4.2 **Denitrification**

The second stage in biological nitrogen removal is called denitrification. In this process, a series of biological reactions are performed to convert the nitrate to nitrogen gas. To oxidize the organic and inorganic electron donors during the denitrification process, nitrite and nitrate play the role of the electron acceptor.

Denitrification can be achieved by different types of bacteria (heterotrophic and autotrophic bacteria), and similar microbial capability has also been found in algae or fungi. Some of the heterotrophic bacteria that accomplish the denitrification are facultative aerobic organisms that can use oxygen with nitrite or nitrate. In addition, a few of these heterotrophic bacteria can achieve fermentation without the need for oxygen or nitrate (Metcalf and Eddy, 2014).

Converting the nitrate to nitrogen gas requires a series of reaction steps from nitrate to nitrite, to nitric oxide, to nitrous oxide, and to nitrogen gas as shown in Equation 2.5.

$$NO_3^{-} \longrightarrow NO_2^{-} \longrightarrow NO \longrightarrow N_2O \longrightarrow N_2 \tag{2.5}$$

Biodegradation of COD in the wastewater as shown in (Equation 2.6), nitrate is the source of the electron donor that is needed for the denitrification. In addition, the denitrification bacteria can acquire the electron donor by the endogenous decay or an exogenous source such as methanol (Equation 2.7) or acetate (Equation 2.8).

$$10NO_3^- + C_{10}H_{19}O_3N \longrightarrow 5N_2 + 10CO_2 + 3H_2O + 10OH^- + NH_3 \quad (2.6)$$

$$6NO_3^- + 5CH_3OH \longrightarrow 3N_2 + 5CO_2 + 7H_2O + 6OH^- \quad (2.7)$$

$$8NO_3^- + 5CH_3COOH \longrightarrow 4N_2 + 10CO_2 + 6H_2O + 8OH^- \quad (2.8)$$

The term $C_{10}H_{19}O_3N$ is often referred to as the biodegradable organic matter in wastewaters.

Although biological systems can treat a large number of the organic carbons, obstinate components are not completely removed. PRW contains a large number of obstinate components (Chavan and Mukherji, 2008); thus, it is hard to biodegrade them completely by biological treatment. This can be detected by the measurement of high COD concentrations in the PRW effluents (Fratila-Apachitei et al., 2001). The remaining COD in the PRW effluent refers to non-biodegradable pollutants (Shokrollahzadeh et al., 2008). Sequencing batch reactor is considered as an efficient technology, low cost, and flexible method which can be used for petrochemical, petroleum and other different industrial wastewater treatment (Patil et al., 2013).

## 2.5 Sequencing batch reactor

For nitrogen removal, conventional activated sludge process is not considered efficient technology. In addition, ASP requires an extra unit for its sludge treatment because the cycle time is not enough to digest the produced sludge. Due to these disadvantages of ASP, alternatives have been introduced such as sequencing batch reactor technology. In the conventional activated sludge process, the wastewater passes from one unit to the other units on a continuous basis, and it needs more area to build these treatment tanks. While in the SBR system, all these units performed within one tank and it works on the same principle as ASP but in a time sequence, and this makes it require less area (Irvine et al., 1979).

SBR system has been successfully used as an efficient technology for wide range of nutrient removal (Demoulin et al., 1977; Keller et al., 2000) as well as industrial, municipal and hazardous wastes treatment (Hersbrun, 1984; Ng and Chin, 1986).

Sequencing batch reactor is an ASP technology that does not require several tanks for the treatment stages as all of these stages could be performed in one tank as well as there being no returned activated sludge (RAS) that returns to other treatment units. Therefore, SBR can be successfully applied in small industries or small areas (Ileri et al., 2003). SBR has been applied as a treatment system for pharmaceutical and domestic wastewater, with 5 hours treatment cycle, more than 88% COD, 82% BOD, 98% suspended solids, and 96% ammonia removal efficiency were achieved (Ileri et al., 2003). In addition, Abu Hasan et al. (2016), achieved up to 89%, 96% and 92.5% removal efficiency for COD, $NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT via SBR system.

There are many differences between the conventional biological wastewater treatment and sequencing batch technology. One of the most apparent differences is the volume of the treatment reactor, it stays constant in the conventional biological wastewater treatment, while it is varying with time in the SBR system. Operating cost requirements for the conventional biological wastewater treatment could be reduced by 60% by replacing this system with the SBR technology (Chang et al., 2000). The SBR system succeeds due to some facts such as its cost-efficient and simple operating requirements, and also the SBR's microbial system could be easily influenced by providing a convenient environment.

SBR technology obtained wide attention in both industrial application and scientific research. Researchers have been studying the SBR technology extensively for pollutant removal by optimising the SBR conditions to get the optimal operational conditions (Wilderer et al., 2001). The removal efficiency of phosphorus has been enhanced because of the sequence of the anaerobic-aerobic process when the phosphate accumulation happens in the first stage, and phosphate utilization is achieved in the second stage (Dassanyakee and Irvine, 2001). In the United State, the SBR system became an attractive treatment option with around 150 SBRs already in operation (Nicolella et al., 1997). The SBR system can be optimised and modified to achieve biological nutrient removal (BNR), nitrification, carbonaceous oxidation and other toxic pollutant removal.

### 2.5.1 **SBR Operations**

The operation of SBR as can be illustrated as follows (Sutton and Mishra, 1990, 1991):

1. Fill stage: This is the first stage of SBR operation, in which the wastewater is added to the reactor and mixed with the bacterial culture inside the treatment reactor to start the treatment activity. This stage can be controlled by timers or liquid level meters. There are three types of fill modes, which are static, mixed, and react fill. In the static fill, the wastewater is added to the treatment reactor without mixing or aeration. While mixing is provided during the mixed fill. Aerated fill means that the aeration is turned on with or without mixing when the wastewater is added to the treatment reactor.

2. React stage: In this stage, the bacterial culture inside the treatment reactor is given the time to biodegrade the organic matter and the other pollutants by providing a proper environment for the bacterial culture to survive and work effectively. The treatment cycle is already started during the fill stage, and in this stage, the aeration or mixing, or both are provided to complete the required treatment. The length of this stage depends on the wastewater characteristics, and it can be controlled by liquid level meters or timers. Depending on the degree of pollution, the react stage may not be required and the aerated fill stage may be enough for the treatment.

3. Settle stage: The purpose of this stage is to separate the treated water from the microbial culture and solids to prepare the treated water for the next stage.

4. Draw stage: In this stage, the treated wastewater is discharged from the SBR treatment reactor through different methods such as adjustable or floating weirs.

5. Idle stage: The is the last stage in the SBR cycle, and it is used in multi-basin only. The time used in this stage will depend on the following reactor to finish its fill

stage. In addition, some of the sludge (bacterial culture) will be wasted in the idle stage. A typical SBR treatment cycle is shown in Figure 2.2.

One of the SBR advantages is that the denitrification is highly likely to be performed during the fill or react stages as well as during the settle and draw stages.

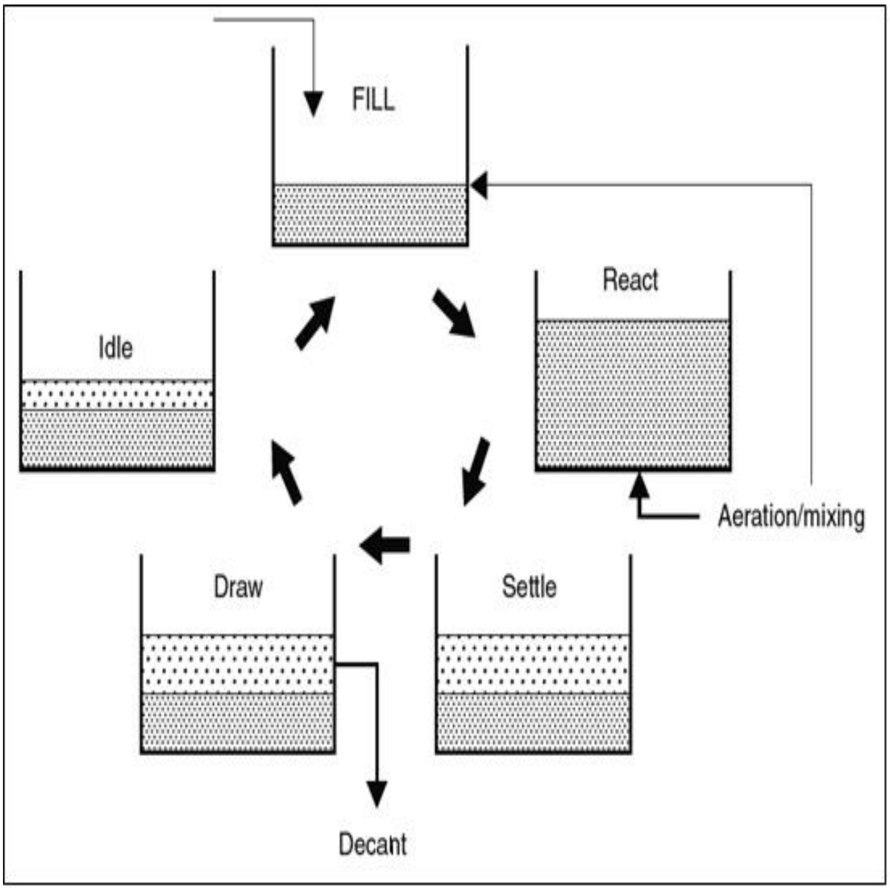



Figure 2.2: Sequencing batch reactor operating cycle
Source (Alattabi et al., 2017b)

To operate the SBR effectively, the quantity of oxygen supplied should be monitored and each cycle time should be set properly without wasting time. Environmental Protection Agency (1999) stated that a typical SBR design criteria as shown in Table 2-3.

Table 2-3: Typical design criteria for SBRs

| Parameter | SBR |
|---|---|
| BOD load (g/d/m$^3$) | 80–240 |
| Cycle time (h) | Variable |
| Fill (aeration) (h) | 1–3 |
| Settle (h) | 0.7–1 |
| Draw (h) | 0.5–1.5 |
| MLSS (mg/L) | 2300–5000 |
| MLVSS (mg/L) | 1500–3500 |
| HRT (h) | 15–50 |
| $\theta_c$ (day) | 20–40 |
| F/M (g $BOD_5$/g MLVSS/day) | 0.05–0.20 |

### 2.5.2 Factors affecting the operation of SBR

#### 2.5.2.1 Mixed liquor suspended solids

MLSS (expressed in milligrams per litre (mg/l)), is the concentration of suspended solids in the mixed liquor. MLSS concentration should be monitored regularly as it can directly affect the treatment efficiency. If its value is high, it will lead to sludge bulking and the treatment system becomes less efficient. Contrariwise, if the MLSS value is low, the energy will be wasted without treating the effluent effectively (Partech, 2016).

Elmolla et al. (2012) operated two SBRs under two different MLSS concentrations (4000 and 6000 mg/l), the results showed that the lower concentration was considered better for the treatment. Tsang et al. (2007) stated that the SBR performance was affected by increasing MLSS concentration as shown in Figure 2.3. However, this disagrees with Martins et al. (2003) who stated that there is no effect of MLSS concentration on the conventional activated sludge process and up-flow aerated biofilter.

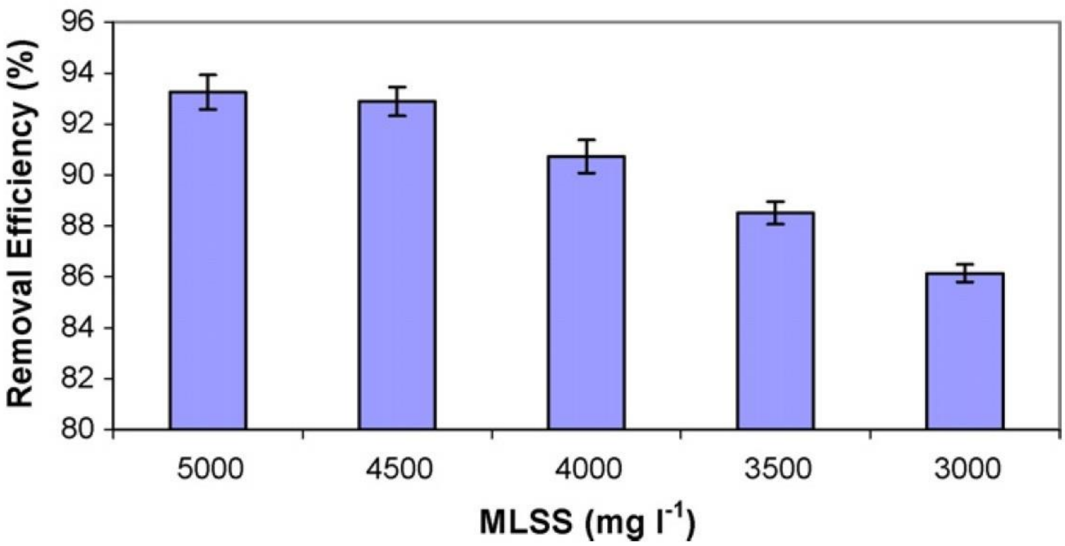



Figure 2.3: COD removal efficiencies under various MLSS

Source (Tsang et al., 2007)

This research project has studied the effects of different concentrations of mixed liquor suspended solids (MLSS) (±2000, and ±3000, ±4000 and ±6000 mg/l) on sludge settleability and effluent quality by studying the sludge characteristics and the removal efficiency for each MLSS concentration in the TSSBR.

#### 2.5.2.2 **Hydraulic retention time**

HRT is one of the most significant parameters in biological treatment as it can affect the degree of treatment of the important pollution parameters. Leong et al. (2011)

stated that via SBR, complete phenol removal had been reached with a 12 h cycle. In addition, Thakur et al. (2013b) studied the effect of HRT and filling time on simultaneous biodegradation of Phenol, Resorcinol and Catechol. Figure 2.4, shows that an increase in HRT from 0.625 d to 1.25 d caused an increase in the COD, phenol, resorcinol and catechol removal efficiencies.

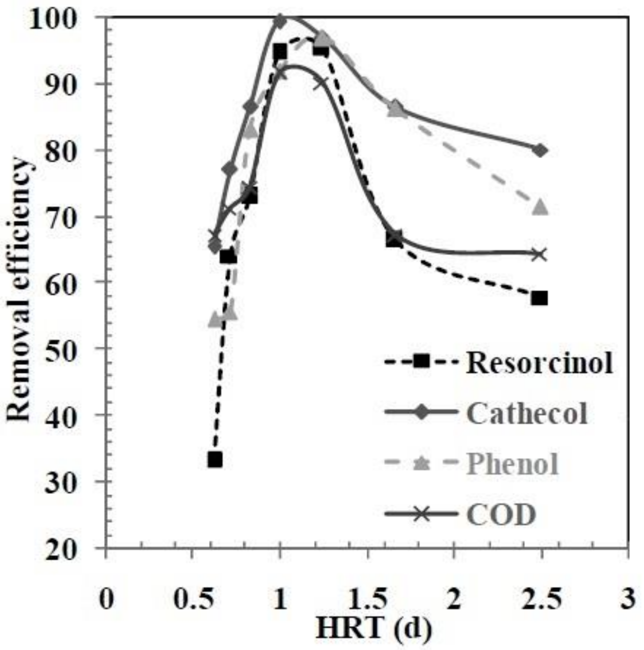



Figure 2.4: Effect of hydraulic retention time (HRT) on the removal of resorcinol, catechol, phenol and COD at SRT= 20 d, instantaneous filling

Source (Thakur et al., 2013b)

Moreover, Thakur et al. (2014) used SBR to reduce the organic matter present in petroleum refinery wastewater, a variation of HRT (0.56-3.33d) was used under instantaneous fill mode as shown in Figure 2.5, the removal efficiency of COD and TOC was 77% and 79% respectively.

Furthermore, SBR with periodic HRT showed better performance than SBR with long HRT (Rodriguez-Caballero et al., 2015). In this research project, the effects of HRT on the sludge settleability and effluent quality in the TSSBR system have been determined by studying different HRTs (4, 6, 8 and 12 hrs).

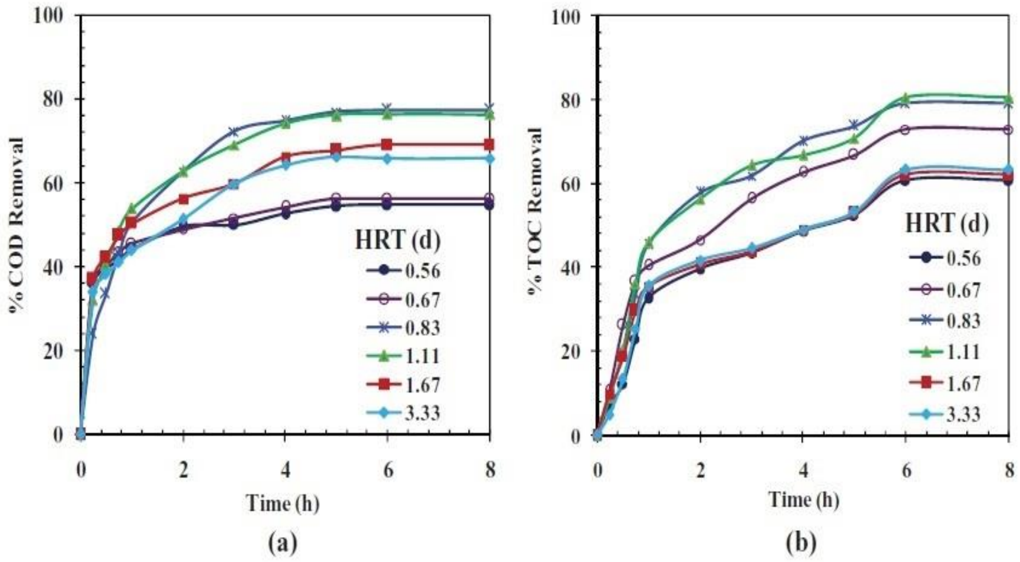



Figure 2.5: Effect of HRT on (a) COD removal. (b) TOC removal
Source (Thakur et al., 2014)

### 2.5.2.3 **Fill conditions**

The fill stage means adding the wastewater to the treatment reactor, and it can be static, mixed or react fill. The time for this stage is variable, and it depends on the wastewater characteristics. Miao et al. (2015) stated that SBR with an aerated fill had been widely used for nitrogen removal in wastewater. Moussavi et al. (2010) examined the performance of aerobic granular SBR to treat phenolic wastewater with different fill time ranging from 1 hour to 4 hours as shown in Figure 2.6, the results showed a decrease in the removal efficiency of phenol from 99.6 to 99% after decreasing fill time from 4 to 1 hour, also it decreased COD removal efficiency from 99 to 97.5%.

In addition, Thakur et al. (2013b) studied the effect of HRT and filling time on simultaneous biodegradation of Phenol, Resorcinol and Catechol, the fill time was varied in the range of 0.5-2 h as shown in Figure 2.7 whereas HRT was kept constant at 1.25 d, the study showed that an increase in fill time from 1.5−2 h reduced the removal efficiency of substrates.

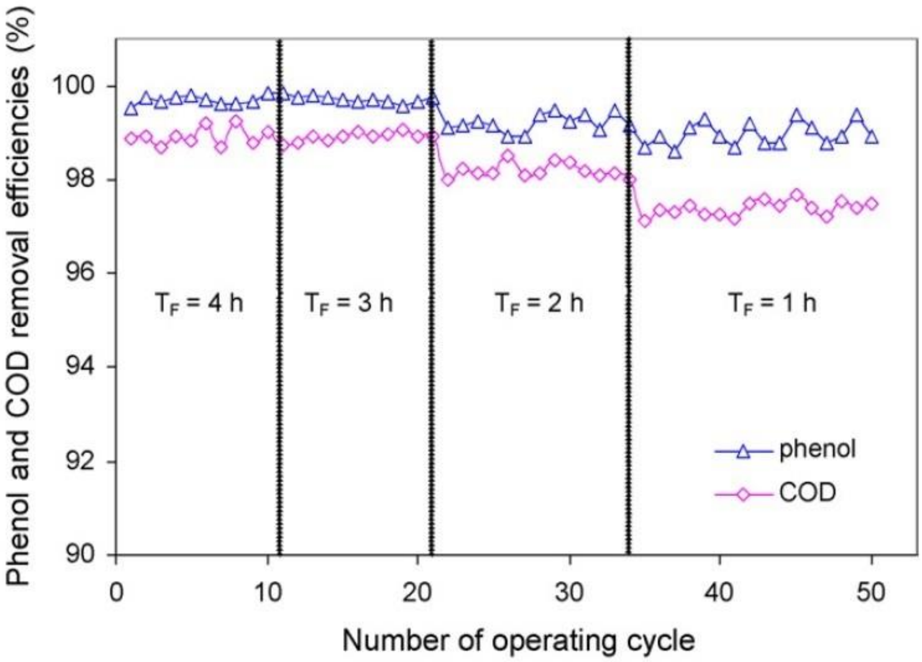



Figure 2.6: Changes of phenol and COD removal efficiencies of the GSBR at different filling times (Tf)
Source (Moussavi et al., 2010)

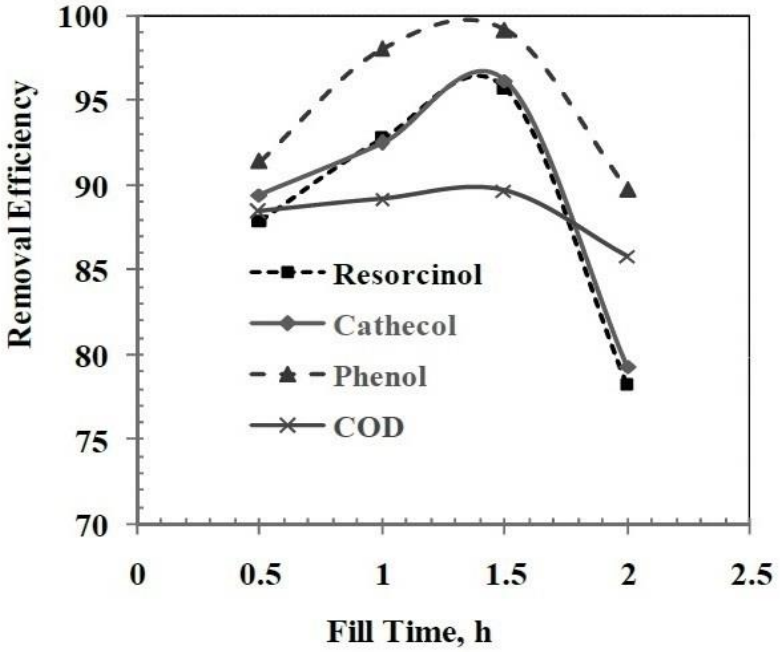



Figure 2.7: Effect of fill time on the removal of resorcinol, catechol, phenol and COD at SRT = 20 d and HRT = 1.25 d
Source (Thakur et al., 2013b)

Following the investigations of (Miao et al., 2015; Moussavi et al., 2010; Thakur et al., 2013b), the fill time range of 15 to 30 minutes proved effective. This study has optimised the fill time between 5 and 30 minutes and studied the sludge settleability and effluent quality for each fill time in the TSSBR as well as the aerated and un-aerated fill mode to determine the effect of fill conditions on sludge settleability and effluent quality.

#### 2.5.2.4 **Organic loading rate**

The organic loading rate is the amount of organic material added to the water. In their study, Moussavi et al. (2010), the effect of initial phenol concentration on the performance of aerobic granular SBR was evaluated as shown in Figure 2.8, it has been noticed that the effect of phenol concentration was insignificant in the range of

100-1700 mg/l, although increasing the concentration of phenol to 2000 mg/l showed a slight decrease in phenol removal efficiency.

In addition, Thakur et al. (2013a) studied the removal of 4-chlorophenol using two SBRs, the first one is blank-SBR without any adsorbent and the second is granular-activated carbon (GAC-SBR), the results showed that the removal efficiency of 4-chlorophenol in GAC-SBR was about 80% for aqueous solutions containing 4-chlorophenol concentration up to 1250 mg/L whereas in blank-SBR the removal efficiency of 4-chlorophenol concentration of 200 mg/L was only 45%. Therefore, compared to blank-SBR, GAC-SBR was able to treat water containing a much higher 4-chlorophenol concentration.

In this research project, the effect of gradually increasing glucose and potassium-nitrate loading rate on the sludge settleability and treatment efficiency of the TSSBR has been studied by investigating four different glucose and potassium-nitrate concentrations.

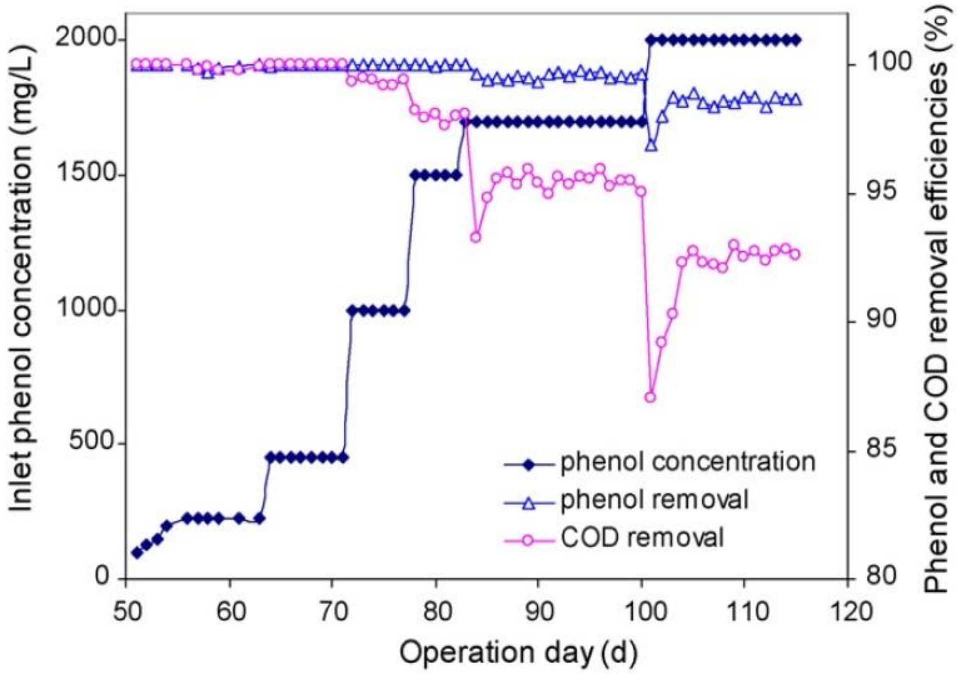



Figure 2.8: Performance of the GSBR in removal of phenol and COD at various inlet concentrations at cycle time of 24 h

Source (Moussavi et al., 2010)

#### 2.5.2.5 **Hydraulic shock**

Shock loading is a sudden or unexpected load that is imposed upon a system. It was employed in a sequencing batch reactor by increasing the influent ammonium concentration from 200 to 1000 mg/l within two months, during the following five months operation period, nitrifying granules exhibited good performance with an ammonium removal efficiency of 99 % (Chen et al., 2015). In addition, Mizzouri and Shaaban (2013) analysed the effects of organic shock loading on SBR in treating PRW; different COD concentrations were applied at varying periods to generate an organic shock as shown in Figure 2.9. The first value of the organic shock load was 0.53 kg COD/kg MLSS d. Such values did not significantly affect SBR performance, COD removal efficiency was 86%, and the effluent TSS was 44 mg/L. While the value of organic shock loading increased to 0.93 kg COD/kg MLSS d. COD removal efficiency was reduced by 68.9%, and the TSS was 64 mg/L.

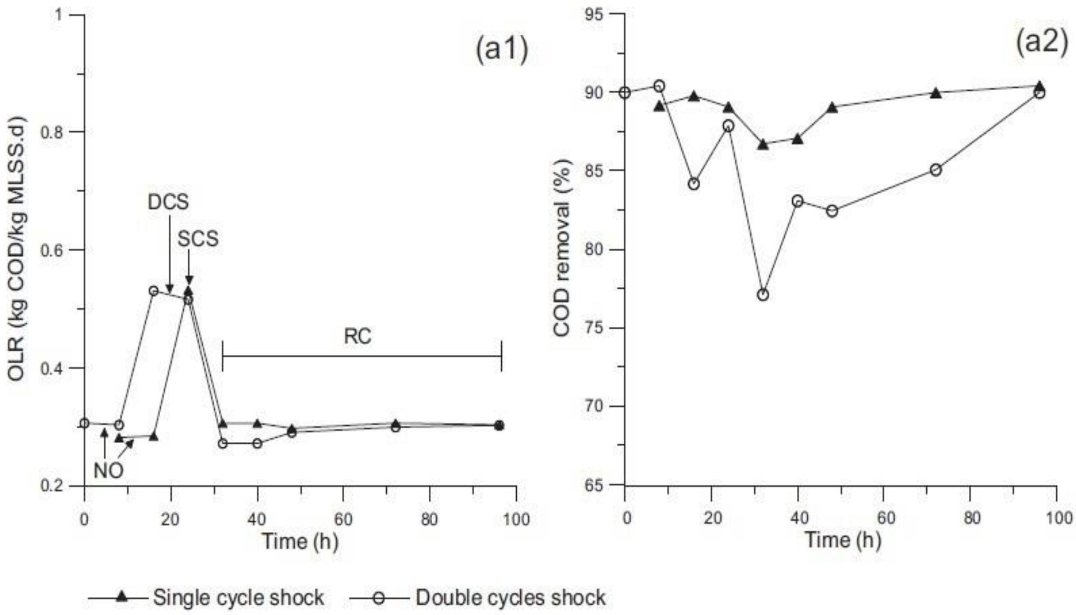



Figure 2.9: Response of SBR system to organic shock loads. NO-normal operation, SCS-single-cycle shock, DCS-double-cycle shock, RC-recovery condition

Source (Mizzouri and Shaaban, 2013)

In this research, the TSSBR capability of handling hydraulic shock has been examined by suddenly decreasing the cycle time of the treatment.

#### 2.5.2.6 **Solids retention time**

Solids retention time (SRT) is the ratio of the mass of solids in the aeration basin divided by the solids exiting the activated sludge system per day. Exiting solids is equal to the mass of solids wasted from the system plus the mass of solids in the plant effluent. Ensuring an adequate SRT is very critical to the SBR biological nutrient-removal design process. The design SRT for nitrifying systems should be based on the aeration time during the cycle, not the entire cycle time (Poltak, 2005).

#### 2.5.2.7 **Sludge wasting**

Sludge wasting should occur during the idle cycle to provide the highest concentration of mixed liquor suspended solids. Sludge from the SBR basins can be wasted to a digester and holding tank for future processing and disposal. The digester-tank and sludge-holding-tank capacity should be sized appropriately, based on the sludge treatment and disposal method. Supernatant from the sludge digester and holding tank should be returned to the headworks or influent equalization basin so that it will receive full treatment. The facility should be designed so that the supernatant volume and load do not adversely affect the treatment process. A high-level alarm and interlock should be provided to prevent sludge-waste pumps from operating during high-level conditions in the digester and holding tanks. Controls should be provided to prevent overflow of sludge from digester tanks and holding tanks (Poltak, 2005).

## 2.6 Online monitoring for nutrient removal

Microbiology activity in the organic matter and nutrient removal involve physical and chemical changes which can be detected through on-line monitoring of pH, dissolved oxygen, (DO) and Oxidation-Reduction Potential (ORP) measurement during a cycle. These changes can give further interesting information for control or process state evaluation. Different critical points can be detected using these relatively simple sensors (pH, ORP and DO) under aerobic or anoxic conditions (Chang and Hao, 1996).

Dissolved oxygen (DO), pH, oxidation-reduction potential (ORP) and alkalinity are variables that must be monitored by the system of SBR. The monitoring of these parameters is so important, and the operator of SBR needs to be able to adjust these variables or to add the chemicals to increase the value of pH and raise the alkalinity to reach the set points. Nitrification process consumes the alkalinity, and that will lead to a decrease in the pH (Slater et al., 2005). Sodium bicarbonate and soda ash are recommended chemicals which can raise the alkalinity and sodium hydroxide can raise the pH. The monitoring of oxidation-reduction potential (ORP) is described in the nitrification and denitrification process where ORP can be used to determine if the chemical reaction is complete or not and can be used to control or monitor the processes.

The operator needs to be able to make some changes in the process by modifying the variable to reach the best removal of undesirable components. The monitoring of dissolved oxygen (DO) is very important in the SBR operation. It allows the operator to adjust the blower times to address the variable organic loads that enter the system, where the monitoring of (DO) can be used to adjust the aeration-blower runtime during the process, which may help to reduce the cost of aeration energy. Generally, the parameters of pH, DO and ORP are monitored online to determine the variations of these variables in the denitrification process, nitrification process, phosphorus release and uptake during aerobic, anaerobic and anoxic phases (Tanwar et al., 2008).

The management of both pH and alkalinity are critical to the effective operation of an SBR. Sufficient alkalinity must be present to allow complete nitrification and result in a residual of at least 50 mg/L in the decanted effluent. The pH must be maintained in a manner to prevent it from falling below 7.0 in the reactor basin. Based on the characteristics of the wastewater, designers should carefully consider the need for both alkalinity and pH management.

ORP measures the electrical potential required to transfer electrons from one compound or element to another compound or element. ORP is measured in millivolts, with negative values indicating a tendency to reduce compounds or elements and positive values indicating a tendency to oxidize compounds or elements. It is desirable to locate DO, pH, and ORP probes in a place that can be reached easily by operators. These probes often clog or foul and need cleaning and calibration. If they are not easily accessible, proper maintenance may not occur (Poltak, 2005).

For plants that nitrify and denitrify, ORP monitoring is desirable. ORP is the measure of the oxidizing or reducing capacity of a liquid. ORP can be used to determine if a chemical reaction is complete and to monitor or control a process. Operators need the ability to make changes that will modify these readings to achieve appropriate nutrient removal. ORP readings have a range and are site specific for each facility. General ranges are: carbonaceous BOD (+50 to +250) mV, nitrification (+100 to +300) mV, and denitrification (+50 to -50) mV (Poltak, 2005).

On-line dissolved oxygen meters are very useful in SBR operation. They allow operators to adjust blower times to address the variable organic loads that enter the plant. Lack of organic strength reduces the react time during which aeration is needed to stabilize the wastewater. DO probes can be used to control the aeration-blower run time during the cycle, which in turn reduces the energy cost of aeration.

### 2.6.1 pH monitoring

The change in pH value during a cycle of a biological system responds to microbial reactions, and hence the pH variation often provides a good indication of ongoing biological reactions. For example, increase in pH for ammonification and denitrification and decreases in pH owing to nitrification. Different critical points can be detected in the pH curve as shown in Figure 2.10 and Figure 2.11. The pH is affected by the stripping of $CO_2$, and as a consequence, an increase of pH occurs as shown in Figure 2.10 (Chang and Hao, 1996).

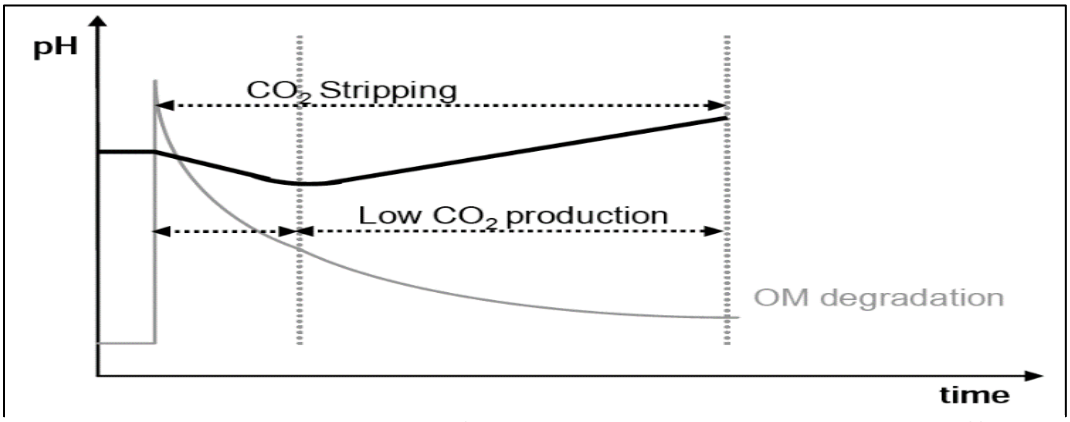



Figure 2.10: Dynamic evolution of pH showing the critical point in the different phases
Source (Chang and Hao, 1996)

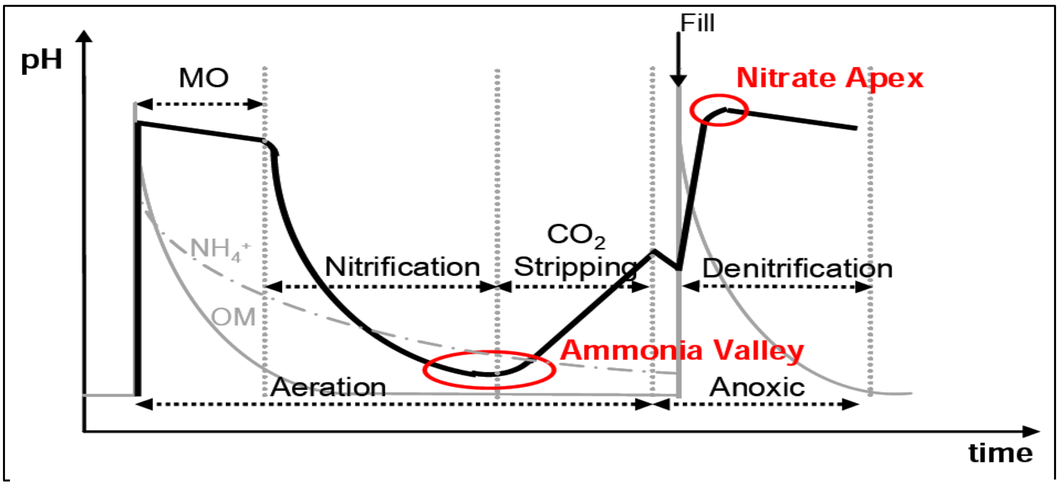



Figure 2.11: Dynamic evolution of pH showing the critical point in the different phases
Source (Chang and Hao, 1996)

The pH profile indicates the properties of the anaerobic phase in biological respiration of the process (Tanwar et al., 2008), and the change in pH profile is basically due to the nitrification and denitrification process which took place in the basin of SBR. The value of pH continuously decreases during nitrification reaction and increases after complete nitrification, and pH values also decrease with the decrease in alkalinity in the reactor. The pH reading decreases at nitrification and increases with carbon-dioxide ($CO_2$) stripping in the aeration phase (Andreottola et al., 2001).

Furthermore, the value of pH for biological plant responds to the microbial activities, and the variation of pH provides a very good indication for the current biological reactions. Also, the monitoring of pH provides further insight into the process dynamic. The readings of pH and ORP together used to adjust the period of the stages in the biological treatment process, and that will lead to providing the process stability (Chang and Hao, 1996).

The studies have shown the effects of pH on nitrification, where the nitrification process is so sensitive to pH of the medium and the optimum pH range for nitrification is 7.5-9.0, if the pH value is out of this range the nitrification process will be decreased sharply. On the other hand, the practical investigation indicates that pH should be controlled carefully in denitrification and phosphorus removal processes in SBR, because the denitrification may increase the pH value in treatment systems and that leads to chemical precipitation of phosphorus (Tyagi and Surampali, 2004).

pH profile can be used as a controlling factor in many SBR operations, where the profile of pH could distinguish the conditions of reaction. The pH profile cannot be used effectively as a control parameter in some cases because it was observed that the pH is ineffective for anoxic phase control (Akin and Ugurlu, 2005).

Marsilli-Libelli (2006) used a laboratory SBR and proposed a switching strategy based on the indirect observation of process state through simple physicochemical measurements and use of a fuzzy inferential engine to determine the most appropriate switching schedule. In this way, the duration of each phase is limited to the time strictly necessary for the actual loading conditions. The experimental results showed that the treatment cycle could be significantly shortened, with the result that more wastewater can be treated.

### 2.6.2 **ORP monitoring**

The oxidation-reduction potential is a measure of the oxidative state in an aqueous system and can be a useful tool for indicating the biological state of a system. ORP elevation is closely related to the dissolved oxygen profile, under aerobic condition. The ORP curve rises with the aeration until an inflection point. This critical point is called α and means that the nitrification is completed (Kishida et al., 2003) as shown in Figure 2.12.

Under anoxic conditions, the ORP profile decreased until the inflection point. This point is called Nitrate Knee and corresponds to the elimination of accumulated nitrate and nitrite (Paul et al., 1998).

The ORP profile provides good information about the process in anoxic phase. The ORP profile is very effective for anoxic phase control while the pH profile is ineffective for anoxic phase control (Akin and Ugurlu, 2005).

In the anoxic phase, the nitrate will be depleted, therefore the change in ORP profile is related to nitrate and that illustrates the end of the denitrification process and total disappearance of nitrate. The aerobic, anaerobic and anoxic phases can be distinguished by ORP profile in the treatment system (Puig et al., 2005).

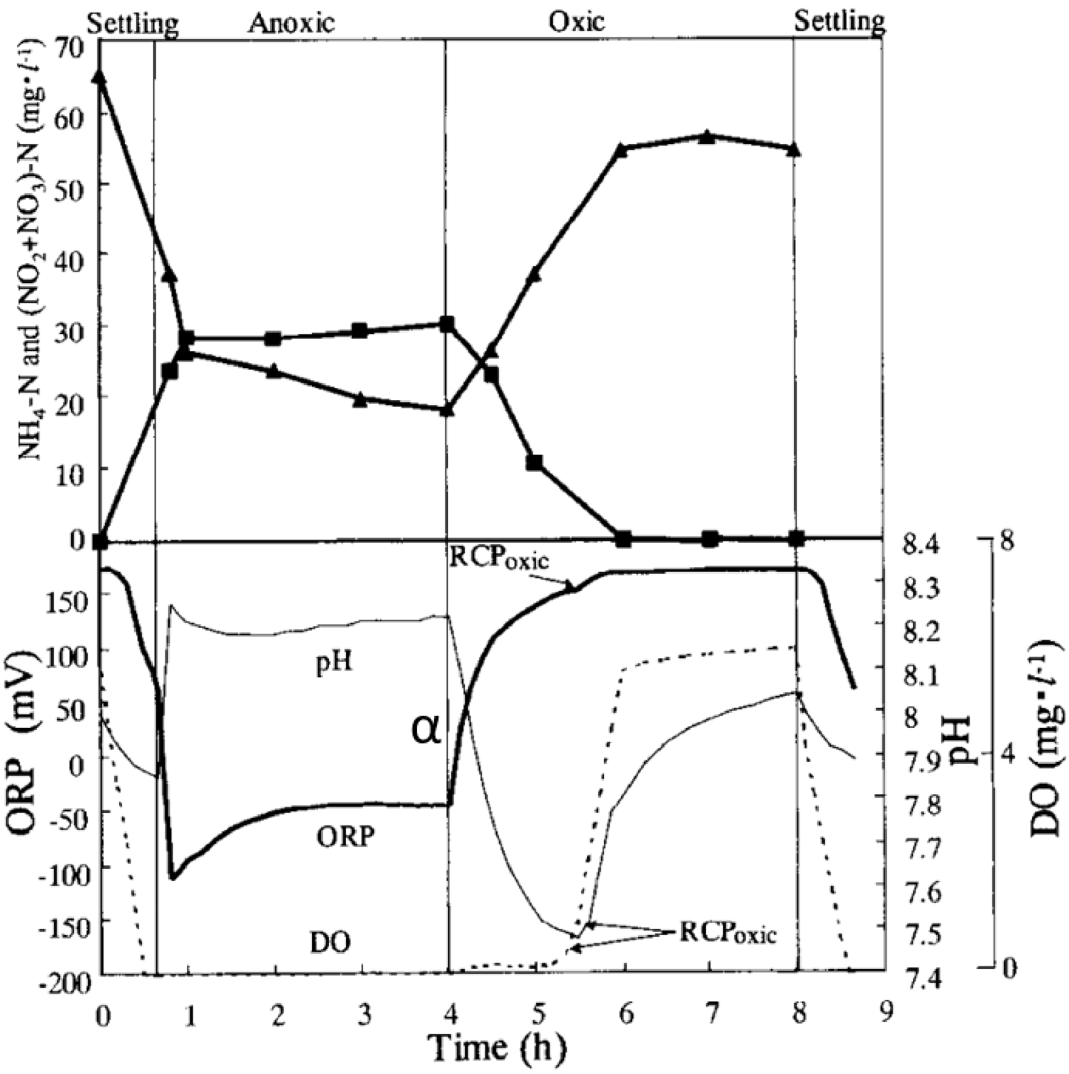



Figure 2.12: ORP and DO profile, under aerobic condition

### 2.6.3 **DO monitoring**

The change in the dissolved oxygen curve responds to microbial reactions; microorganisms utilize oxygen as an electron acceptor under aerobic conditions. Under aerobic filling phases, the organic carbon oxidation is very high and requires a large quantity of oxygen which causes the DO to decline to a low level in the reactor.

When organic matter is close to being completely removed, a sudden DO increase is observed. Afterwards, the main reaction is the oxidation of ammonia (nitrification), and here the DO rises progressively.

The DO profile can be used in the oxic phase only when the value of DO is above zero. Because of the inability to monitor the DO values in anoxic or anaerobic phase. The previous studies have shown that the nitrification is completed when the DO values are less than 0.5 mg/L at short sludge ages (Akin and Ugurlu, 2005). The studies also show that many heterotrophic bacteria have the ability for nitrification, where the heterotrophic bacteria can grow faster than the autotrophic at low levels of DO (Zhao et al., 1999). The rate of nitrification is higher when the level of oxygen is low, and this may illustrate that the heterotrophic bacteria for nitrification can be present in the reactor of treatment dominantly. The nitrification is inhibited when the value of DO is more than 1.0 mg/L (Chuang et al., 1997).

Akin and Ugurlu (2005) monitored the profiles of pH, DO and ORP to establish control strategies for biological phosphorus and nitrogen removal. They used a laboratory scale SBR system and found that pH and ORP values can be used as control parameters for denitrification and biological phosphorus removal. However, it is observed that the pH profile provides much information during the oxic phase, whereas ORP does in the anoxic phase.

### 2.6.4 **Temperature monitoring**

Temperature is considered an important parameter which can affect the water treatment performance as well as the power generation. It may affect the bacterial kinetics and the types of bacteria that survive in the treatment reactor. Therefore, while bacterial growth rate and respiration can be affected by the changes in temperature, the bacterial community development and bacterial structure can also be affected by temperature changes (Tee et al., 2017).

Wastewater temperatures could drift due to seasonal changes. Gradual temperature variations may affect the microbial community structure in biological wastewater treatment, and sudden temperature changes may also affect negatively on the process performance. It is an expensive procedure to change the temperature of wastewater for biological treatment at an industrial scale. Some industrial wastewaters may be cooled down to suitable levels for biological treatment, but wastewaters are not typically heated, nor their temperature controlled because of the high expense that this would entail. (De Grazia et al., 2017). Therefore, the temperature of the treatment reactors should be monitored and relate the temperature effect on the bacterial growth in the system.

## 2.7 Solid settling problems

It has been reported that SBRs require less area, are flexible to operate and can be operated automatically (Abu Hasan et al., 2016; Alattabi et al., 2017b). However, solid-liquid separation, or sludge bulking, is still one of the most problematic issues with SBRs and ASPs in general (Guo et al., 2014b; Koivuranta et al., 2015).

Researchers have reported several reasons for this problem such as difficulty in handling sudden changes in the operating parameters (Alattabi et al., 2017d; Alattabi et al., 2016), microbial clustering behaviour (Ye et al., 2016), the overgrowth of filamentous bacteria (Martins et al., 2004; Mesquita et al., 2011), foaming (Guo et al., 2014a; Guo et al., 2012), pin-point sludge (Guo et al., 2012; Jenkins et al., 2003), poor macrostructure (Guo et al., 2012), poor flocculation properties (Jin et al., 2003) and floc size distribution (Amaral and Ferreira, 2005; Mesquita et al., 2011).

Settleability problems and loss of solids in activated sludge processes may be due to one operational condition, such as the undesired growth of filamentous organisms, or several operational conditions, for example, the undesired growth of filamentous organisms and the presence of nutrient-deficient floc particles and foam. Some operational conditions frequently occur in many activated sludge processes and receive many reviews in the literature. These were frequently occurring operational conditions include the undesired growth of filamentous organisms, nutrient-deficient floc particles, and denitrification. Several operational conditions occur infrequently in activated sludge processes and receive little review in the literature, examples of these conditions include cell bursting agents, elevated temperatures, and colloidal floc particles (Gerardi, 2002).

### 2.7.1 **Factors causing solid settling problems**

#### 2.7.1.1 **Undesired filamentous growth**

Filamentous organisms are chains of microscopic cells. There are approximately 30 filamentous organisms that are commonly found in activated sludge processes. Most filamentous organisms are usually 50–1000 mm in length and are straight, curved, or coiled in shape. Filamentous organisms may be found within the floc particles, extending into the bulk solution from the perimeter of floc particles, and free-floating in the bulk solution.

Filamentous organisms enter activated sludge processes in relatively large numbers of individual cells, short chains of cells, or broken chains from a variety of sources.

Filamentous organisms are common soil and water organisms that enter an activated sludge process. They grow in the biomass covering the bottom of manholes and the inside of sewer mains and are continuously washed into activated sludge processes as wastewater flows over the biomass. Industries that use biological processes to pre-treat their wastewater before it is discharged to a municipal sewer system may discharge filamentous organisms in their effluent.

Three groups of filamentous organisms affect the operation of an activated sludge process. These organisms are algae, bacteria, and fungi. Most filamentous organisms are bacteria. The bacterial group includes the Nocardioforms that are best known for their production of viscous, chocolate-brown foam on the surface of an aeration tank and collapsed foam (scum) on the surface of secondary clarifiers. Examples of Nocardioforms include Nocardia amarae and Nocardia pinensis (Gerardi, 2002).

Filamentous organism foam such as that produced by Nocardioforms is typically viscous and chocolate-brown. Active and dead cells produce the foam. Active cells release lipids that coat the floc particles and capture air bubbles and gases, and dead cells release biosurfactants that reduce the surface tension of the wastewater. The major biosurfactants released are ammonium ions and fatty acids.

When filamentous organism foam enters the secondary clarifier, entrapped air bubbles and gases are released as the foam spills over the influent weirs of the clarifier. The escape of air bubbles and gases causes the foam to collapse. The collapsed foam is often referred to as scum (Gerardi, 2002).

One of the most common problems in the activated sludge process is filamentous bulking, affecting most treatment plants working on the activated sludge principle. A bulking sludge can be defined as the sludge that compacts and settles slowly. Usually, in the treatment plants, it can be considered as bulking sludge if the SVI value is greater than 150 ml/g.

However, SVI value can vary from one treatment plant to another, and they would not have the same settling behaviour even if the SVI values were the same, due to the differences in the size and efficiency of the final clarifier(s) and hydraulic considerations. Therefore, a bulking sludge may or may not lead to a bulking problem, depending on the specific treatment plant's ability to contain the sludge within the clarifier.

Growing a certain amount of filamentous bacteria in the activated sludge process can be beneficial to the system. On the other hand, a lack of filamentous bacteria in the activated sludge process might lead to small, easily sheared flocs (pin-floc) that has a good settling ability but it could leave behind a turbid effluent. Filaments are very important to floc structure, helping the formation of stronger, larger flocs. The presence of a certain amount of filaments also helps to catch and hold small particles through sludge settling, yielding a lower turbidity effluent. However, it would affect negatively on the sludge settleability, only if the filaments grow in large amounts.

Two basic forms of interference in sludge settling occur, depending on the type of filament: the first form of interference called “open-floc structure” in this type, the filaments grow mostly within the floc, and the floc grows around and attaches to the filaments. In this type of interference, the floc becomes irregularly-shaped, large, and contains substantial internal voids. The second form of interference is called “interfloc-bridging" in this type, the filaments extend from the floc surface and physically hold the floc particles apart.

A bulking sludge can cause serious environmental damage and affect negatively on the effluent quality by losing the sludge inventory to the effluent. In severe cases, a loss of the plant's treatment capacity and failure of the process could occur because of the loss of the sludge inventory. In addition, disinfection of the treated effluent can become more complex by the excess solids present in the effluent during bulking. In less severe cases, bulking sludge leads to excessive return sludge recycle rates, and this could cause problems in waste activated sludge disposal. Most of the problems in waste sludge thickening are filamentous bulking problems (Richard et al., 2003)

#### 2.7.1.2 **Nutrient-deficient floc particles**

A nutrient deficiency in an activated sludge process may result in several operational problems (Table 2-4). These problems include loss of settleability, loss of solids, and the production and accumulation of foam. The nutrient deficiency usually is for nitrogen or phosphorus and most often is associated with the discharge of industrial wastes that are rich in soluble cBOD but lacking in proper quantity and quality of at least one nutrient. Because industrial wastes are usually responsible for a nutrient deficiency in an activated sludge process, the occurrence of a nutrient deficiency may be examined with respect to the type of wastewater that is treated and the type of nutrient that may be deficient (

Table 2-5) (Gerardi, 2002).

Table 2-4: Operational problems associated with a nutrient deficiency
Source (Gerardi, 2002)

| |
|---|
| Decreased cBOD removal efficiency |
| Foam production and accumulation |
| Decreased nBOD removal efficiency |
| Lack of adequate MLVSS production |
| Loss of solids |
| Settleability problems |
| Undesired growth of nutrient deficient filamentous organisms |

Table 2-5: Nutrients required by all bacteria
Source (Gerardi, 2002)

| | |
|---|---|
| Major nutrients: | C, Ca, Cl, H, K, N, Mg, Na, O, P, S |
| Minor nutrients: | B, Co, Cu, Cr, F, Fe, I, Mn, Mo, Ni, Se, Si, V, Zn |

#### 2.7.1.3 **Low dissolved oxygen concentration**

A low dissolved oxygen concentration in the aeration tank may be associated with several operational problems. Low dissolved oxygen may be associated with the undesired growth of filamentous organisms (Table 2-6), loss of treatment efficiency for cBOD removal, loss of treatment efficiency for nBOD removal or nitrification, and the interruption of floc formation.

It is not the absence of dissolved oxygen that causes the interruption of floc formation, but the presence of a low dissolved oxygen concentration. This concentration hinders proper floc formation. Dissolved oxygen values responsible for the interruption of floc formation and loss of fine solids are <1.0 mg/l for ten or more consecutive hours.

A low dissolved oxygen level contributes to the interruption of floc formation and loss of solids through two significant and detrimental changes in the biomass. First, and more importantly, the floc bacteria are adversely affected. Second, the ciliated protozoan population is damaged (Gerardi, 2002).

Table 2-6: Filamentous organisms that proliferate under a low dissolved oxygen concentration
Source (Gerardi, 2002)

| |
|---|
| Haliscomenobacter hydrossis |
| Microthrix parvicella |
| Sphaerotilus natans |
| Type 1701 |

### 2.7.1.4 **Temperature**

Temperature has a significant impact on the activity of all organisms in the activated sludge process and the development and settling character of floc particles as shown in Figure 2.13. This impact causes physical and biological changes that affect floc particle structure and the settling rate of secondary solids.

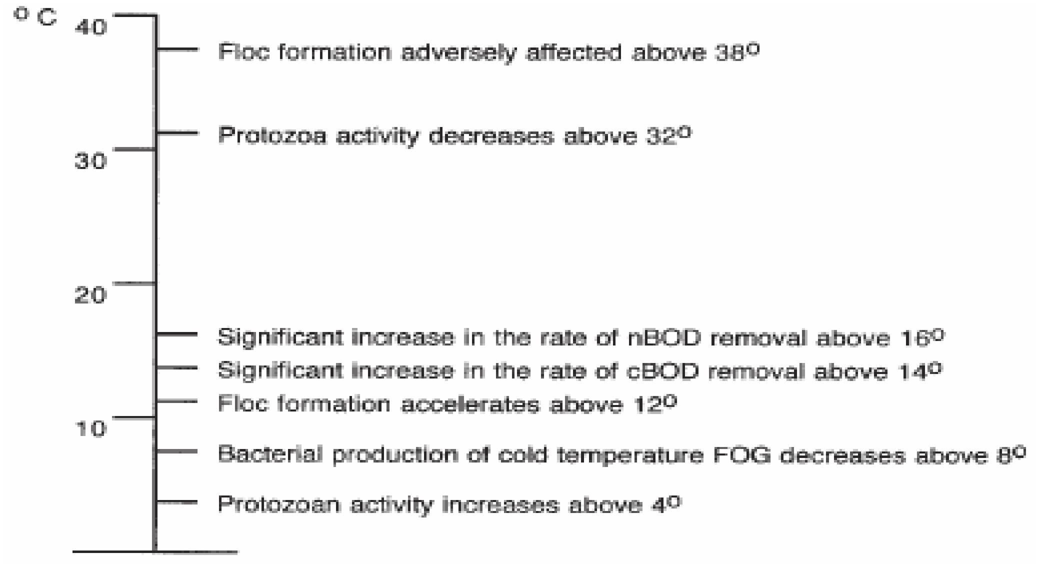



Figure 2.13: Impact of temperature upon the activated sludge process
Source (Gerardi, 2002)

#### 2.7.1.4.1 Physical changes

As wastewater temperature becomes colder, the wastewater becomes denser. Therefore, the settling rate of secondary solids decreases. However, the physical impact of cold temperature on the settling rate of secondary solids is not significant unless the MLVSS is relatively high, for example, >10,000 mg/l.

As wastewater temperature becomes warmer, the wastewater becomes less dense. Therefore, the settling rate of secondary solids increases in warm wastewater temperature. Again, the physical impact of warm temperature on secondary solids is not significant unless the MLVSS is relatively high.

#### 2.7.1.4.2 Biological changes

The impact of biological changes that affect the floc particle structure and rate of settling of the secondary solids that are caused by changes in wastewater temperature occurs at relatively small MLVSS concentrations, for example, 2000mg/l. The changes in the settling rate of secondary solids caused by changes in wastewater temperature are opposite to those changes caused by physical changes.

With increasing wastewater temperature, bacterial activity increases. Increased production and accumulation of insoluble biological secretions such as lipids and oils accompany this increase in activity. These secretions are adsorbed or entrapped by the floc particles, resulting in a decreased settling rate of secondary solids. When air bubbles or gases become entrapped in these secretions, the settling rate of the secondary solids decreases more.

With decreasing wastewater temperature, bacterial activity decreases. Decreased production and accumulation of insoluble biological secretions and a decreased number of entrapped air bubbles and gases accompany this decrease in activity. Therefore, the settling rate of the secondary solids is not as slow during decreasing wastewater temperature compared with increasing wastewater temperature (Gerardi, 2002).

Changes in wastewater temperature have a significant impact on the activity of all organisms, floc particle structure, and the rate of floc formation.

### 2.7.2 **Current solutions for settling problems**

#### 2.7.2.1 **Granulation technology**

In a specific environment, microbial self-agglomeration forms a granular biological polymer which is known as aerobic granular sludge (AGS) (Kreuk et al., 2007; Long et al., 2016). It has many advantages such as high degradation ability, significant settling velocity, regular shape and compact structure (Adav et al., 2008a; Chen et al., 2013; Long et al., 2016; Show et al., 2012; Zhang et al., 2015).

Chen et al. (2013) used granular sequencing batch reactors (GSBRs) to enhance nitrogen removal, the system showed outstanding performances with over 85% nitrogen removal efficiency and good settling ability with SVI of 20 ml/g in treating wastewater with C/N ratio of 5:1. Long et al. (2016) used aerobic granules for the treatment of solvent in a bench scale sequencing batch reactor, the results showed that aerobic granular sludge became stable after 55 days and it could treat high C/N ratio industrial wastewater, and a good removal effect could be achieved. Zhang et al. (2015) used aerobic granular sludge in an SBR; the results showed that the removal efficiencies of COD, $NH4^+$–N, total nitrogen (TN) and phosphate (P) reached 99%, 98%, 90% and 99%, respectively.

However, AGS stability might decline after a long period of operation (Adav et al., 2008b; Lee et al., 2010; Liu and Liu, 2006; Liu et al., 2004; Tay et al., 2002). In addition to the stability loss, granulation technology has other problems such as producing high operation temperature, needing long acclimatisation time and not being efficient with a low concentration of organic wastewater (Lettinga et al., 1980; Qin et al., 2004), which makes granulation technology need more research to tackle these issues.

#### 2.7.2.2 **Polymer and coagulant addition**

Another attempt to overcome the settling problem is chemical addition before the settling stage to improve the settling performance (Agridiotis et al., 2007; Wu et al., 1997)

To improve the settling of the activated sludge process, different types of chemical could be added to the ASP (Agridiotis et al., 2007). One popular chemical is synthetic, high molecular weight, anionic polymer, and this could be added alone or in combination with cationic polymers that serve to overcome the physical effects of filaments on sludge settleability. The chemicals are usually added to the biomass that is leaving the aeration tank or to the secondary clarifier. Use of polymer does not significantly increase waste sludge production but can be quite expensive, up to $450 per million gallons treated (Richard et al., 2003). In addition, inorganic precipitants/coagulants such as ferric chloride or lime are added to sweep down the activated sludge, improving settling by producing a voluminous precipitate for this purpose. The addition of these precipitants may significantly increase the sludge production (Richard et al., 2003).

Activated carbon has also been used recently to improve the settling. Activated carbon treatment adsorption can be applied via tertiary granular activated carbon (GAC) columns, or powdered activated carbon (PAC) integrated into the activated sludge process (Hami et al., 2007). Aziz et al. (2011) used powdered activated carbon with an SBR for treatment of landfill leachate; the results showed that the powdered activated carbon SBR exhibited a significant improvement in the treatment of landfill leachate compared with the normal SBR. Thakur et al. (2013a) studied the removal of 4-chlorophenol in a sequencing batch reactor with and without granular-activated carbon, the results showed that cycle time of blank-SBR decreased from 8 to 6h when granular-activated carbon was used as an adsorbent in the SBR, the removal efficiency of 4-chlorophenol was improved from 73% to 96.9% when granular-activated carbon was used as an adsorbent in the SBR. However, adding these chemicals to improve the settleability could raise the cost of treatment and result in more complex and toxic residues which affect negatively on the environment (Iritani et al., 2015).

#### 2.7.2.3 **Chlorination**

Hydrogen peroxide and chlorine have been used successfully to reduce the sludge bulking and eliminate the growth of filamentous bacteria. Because of its availability at most plants as well as its low cost, chlorine has been used widely to control the sludge bulking. The aim of chlorination is to expose the chlorine to the activated sludge to damage filaments extending from the floc surface while leaving organisms within the floc untouched. Floc-forming bacteria and filamentous bacteria showed the same behaviour while exposed to chlorine.

It is noteworthy to highlight that chlorination is not a solution for all activated sludge microbiological problems. Chlorination might increase the problem if there are no filamentous bacteria in the activated sludge, or if the problem was poor floc development. In addition, over chlorination could result in a loss of the higher life forms (protozoa), a significant increase in effluent TSS, and a reduction in BOD removal. It is normal to see a small increase in effluent $BOD_5$ and effluent suspended solids while controlling the sludge bulking by chlorination (Richard et al., 2003).

#### 2.7.2.4 **SBR operational control**

To improve the treatment performance and settleability, researchers have been modifying the operation strategy or adding more stages to the SBR treatment cycle without additional cost if the cycle time did not increase. Aziz et al. (2011) studied two-stage SBR with powdered activated carbon addition, the performance of this system showed an increase in the removal of chemical oxygen demand, colour, and total dissolved salts, improved sludge characteristics, and greater ability to save aeration energy. Chen et al. (2013) used alternating anoxic/oxic condition combined with step-feeding mode to enhance nitrogen removal, this operating procedure resulted in a reduction of extra carbon addition for denitrification, which may greatly broaden its application in practice, especially for wastewater with low C/N ratio, the system showed outstanding performances with over 85% nitrogen removal efficiency and good settling ability with SVI of 20 ml/g in treating wastewater with C/N ratio of 5:1. Mata et al. (2015) studied the effect of the SBR cycle strategy on the treatment of simulated textile wastewater with aerobic granular sludge; he compared between a single aeration phase and intermittent aeration phase. The intermittent aeration cycle strategy led to marked performance improvements, inducing the formation of dense, faster settling aggregates. The overall removal of COD with the intermittent aeration regime has been improved significantly.

Based on the previous statement, comes the aim of this research which is introducing a novel two-stage settling SBR to eliminate the filamentous accumulation and improve the settling stage. In addition, separating the two stages of settling with a short anoxic stage might enhance the nitrogen removal efficiency by improving the denitrification stage.

## 2.8 Chapter summary

This chapter reviewed the petroleum refinery wastewater, its classification and treatment methods including physical, chemical and biological treatment. Then it focused on the biological treatment method as is considered a cheap and efficient method of treatment. Biological nitrogen removal has also been discussed briefly in this chapter. Sequencing batch reactor was selected in this research project because it is an activated sludge process (one of the biological treatment methods) that required less area to operate. SBR operation has been briefly discussed. In addition, the factors affecting the operation of SBR including (mixed liquor suspended solids, hydraulic retention time, fill conditions, organic loading rate, hydraulic shock, solid retention time and sludge wasting) have been briefly discussed in this chapter. Then the online monitoring for nutrient removal including (pH, DO, ORP and temperature) has been briefly discussed. Finally, the solids settling problems have been discussed in two parts, first the factors causing solids settling problems including (undesired filamentous growth, nutrient-deficient floc particles, low dissolved oxygen concentration and temperature) have been briefly discussed, second the current solutions for settling problems including (granulation technology, polymer and coagulant addition, chlorination and SBR operational control) have been briefly discussed.

Therefore, it is proposed that there is an ability to improve the settling problems in the SBR system by a two-stage settling SBR system, which will be discussed in the next chapter (Chapter 3) along with the material and methods that were used in this research.

# CHAPTER 3

# Methodology

## 3.1 Innovative two-stage settling SBR

A novel, two-stage settling sequencing batch reactor separated by a 15 minutes anoxic stage has been introduced in an attempt to improve sludge settleability of the SBR system.

### 3.1.1 Design description

The operating cycle of the two-stage settling SBR is shown in Figure 3.1. Operating the SBR system in this cycle could have a few positives outcomes. Firstly, a shock will be created after the first settling stage to allow small flocs to cling together and merge with larger flocs before settling again in the second settling stage. The effect of this procedure, the elimination of filamentous accumulation and improvements in the settling stage will then be examined. Finally, verification will be sought as to whether separating the two stages of settling with a short anoxic stage, enhances the efficiency of the removal of nitrogen by improving the denitrification stage (Chen et al., 2013; She et al., 2016).

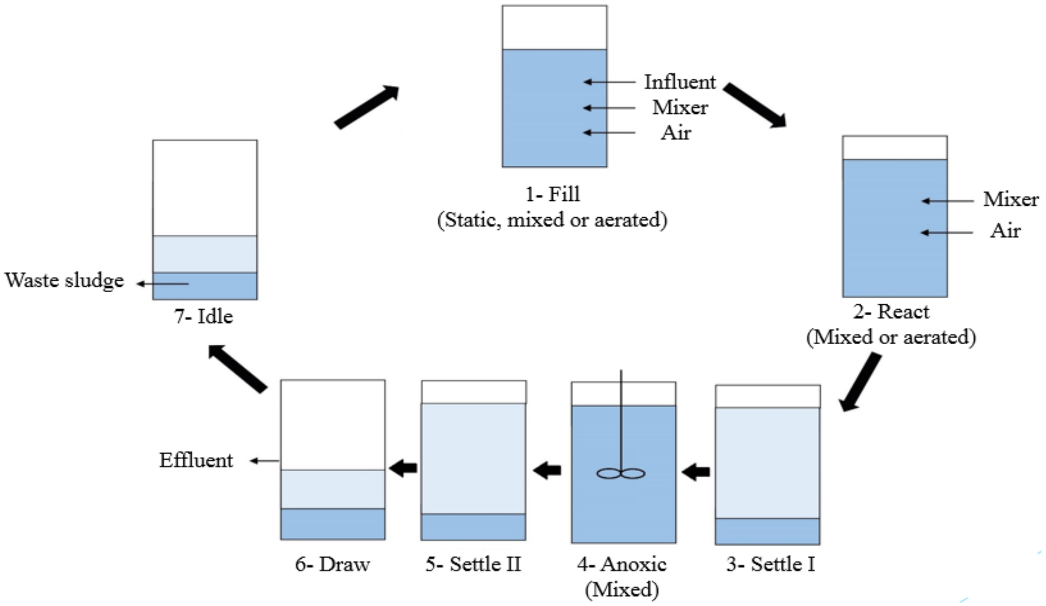



Figure 3.1: The operating cycle of the two-stage settling SBR

Source (Alattabi et al., 2017c)

### 3.1.2 **Cost comparison**

The difference between the normal SBR and the two-stage settling SBR is the settling stage, which is 60 minutes settling stage for the normal operating SBR, while there are two settling stages (15 minutes and 30 minutes) separated by 15 minutes anoxic mixing stage for the two-stage settling SBR. Thus, the total cycle time for both systems is the same. The two-stage settling SBR has 15 minutes mixing stage each cycle of treatment which requires more power to operate, and this should be considered when comparing the results of the two systems to see if it is worthy to operate this system in the industry or not.

## 3.2 Synthetic wastewater and laboratory setup

### 3.2.1 Introduction

SBR system has the ability to treat different types of wastewater by microbial activities to improve the quality of water before its disposal to the water bodies. This study was carried out using SBR technology to treat the synthetic wastewater containing a complex of chemicals. The parameters of COD, $NH_3$-N, $NO_3$-N, $NO_2$-N and SVI were measured periodically to study the SBR performance. Also, the study was carried out to evaluate SBR performance by daily monitoring of the parameters; dissolved oxygen, oxidation-reduction potential, temperature and pH.

### 3.2.2 Activated sludge characteristics and synthetic wastewater

The activated sludge used in this study was obtained from a wastewater treatment plant called Sandon Docks, located in Liverpool, UK. Synthetic wastewater was used in this study rather than real wastewater due to health and safety requirements in the LJMU labs. Influent synthetic wastewater was prepared in deionised water, as shown in Table 3-1. Glucose was used as the carbon source, Ammonium Chloride and Potassium Nitrate used as ammonia and nitrate sources, Magnesium Sulphate Heptahydrate and Monobasic Potassium Phosphate used as phosphate sources, Sodium Bicarbonate used as a buffer solution to maintain the pH value within 6.5 - 8, the remaining chemicals used as trace elements to represent wastewater (Shariati et al., 2011; Zhao et al., 2011). All reagents used in this study were purchased from Sigma-Aldrich, UK.

Table 3-1: Concentration and compositions of the synthetic wastewater Source (Alattabi et al., 2017c)

| Chemicals | Chemical formula | Concentration |
|---|---|---|
| Glucose | $C_6H_{12}O_6$ | 500 mg/l |
| Magnesium Sulphate Heptahydrate | $MgSO_4.7H_2O$ | 5 mg/l |
| Sodium Bicarbonate | $NaHCO_3$ | 200 mg/l |
| Ammonium Chloride | $NH_4Cl$ | 25 mg/l |
| Potassium Nitrate | $KNO_3$ | 25 mg/l |
| Monobasic Potassium Phosphate | $KH_2PO_4$ | 5 mg/l |
| Iron(III) Chloride Hexahydrate | $FeCl_3.6H_2O$ | 1.5 mg/l |
| Calcium Chloride Dihydrate | $CaCl_2.2H_2O$ | 0.15 mg/l |

### 3.2.3 **Experimental setup and operation of the treatment reactors**

Four identical reactors were used in this study. Each is made of Plexiglas and has a total volume of 6.5L with a working volume of 5L. Peristaltic pumps were used to fill and withdraw the effluent wastewater. Air diffusers were used to supply the reactors with fine air bubbles. Mixing was carried out using an overhead stirrer at a speed of 300 rpm. Four electronic sensors (probes) were installed in each reactor to monitor the pH, oxidation-reduction potential, temperature and dissolved oxygen.

Each SBR reactor was filled with 1.5L of activated sludge and 3.5L of synthetic wastewater. Air was supplied at the rate of 1l/min, pH was maintained between 6.5 and 8, the temperature was between 12-25 C°. To acclimatise the microorganisms, the treatment reactor was aerated for 20 days. Following this, synthetic wastewater was added to the reactor and samples taken and analysed from each treatment reactor for influent and effluent respectively. Besides, new sludge was added to the reactors every 20 days to keep the bacteria active (Ekama, 2010).

The SBR operation was carried out as follows: synthetic wastewater was transferred from the storage tank to the treatment reactors through peristaltic pumps in the first 15 minutes (fill stage), then an influent sample was taken and analysed from each reactor to measure the influent wastewater and to make sure the synthetic wastewater in the storage tank was stable. Aeration was introduced to the reactors for 240 minutes (react stage). Settling is the third stage in the SBR operation, achieved by turning off the aeration and mixing for 0.5-1 hr. The fourth stage, draw or decant, was to discharge the treated wastewater from the reactor via peristaltic pumps, this taking 15 minutes. The idle stage is the last stage where a certain amount of sludge is discharged from the treatment reactor to keep the system under the target concentration of MLSS. The SBR was operated continuously for the whole period of study, sampling and analyses carried out twice a week due to cost consideration.

The configuration of SBR1, SBR2, SBR3 and SBR4, and the whole system of laboratory SBR used in this research is shown in Figure 3.2 and Figure 3.3.

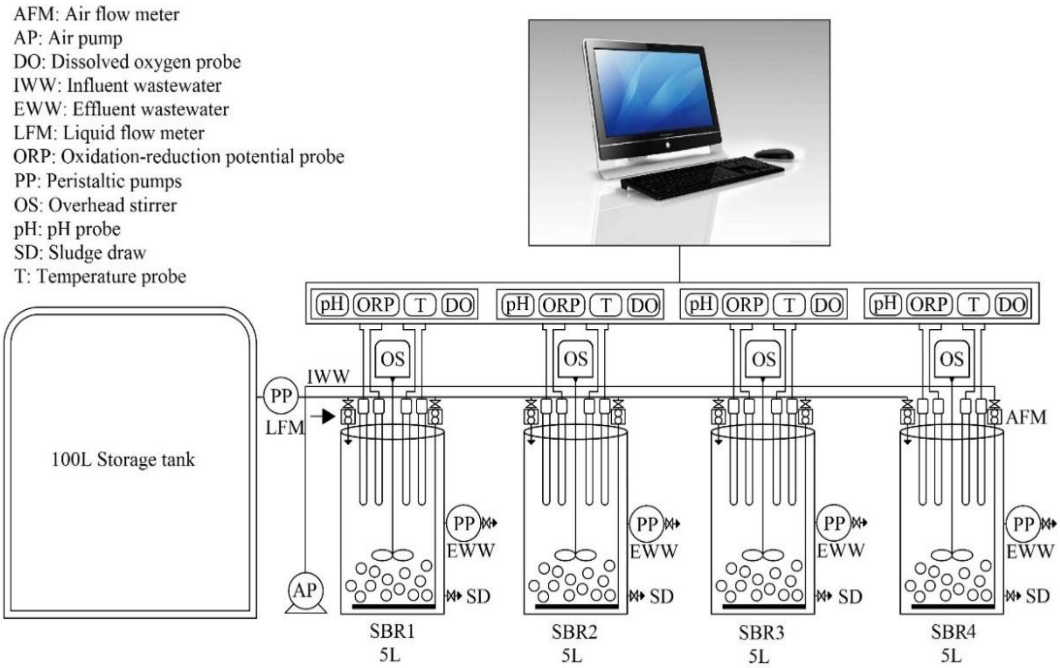



Figure 3.2: The configuration of laboratory SBRs (SBR1, SBR2, SBR3 and SBR4)

Source (Alattabi et al., 2017d)

Figure 3.3: The whole system of laboratory SBR
Source (Alattabi et al., 2017c)

### 3.2.3.1 **Acclimatization of mixed culture**

Mixed liquor suspended solids were studied for all the treatment reactors. The synthetic wastewater in Table 3-1 were added to the reactors and pH, DO, temperature and ORP were monitored online to ensure a good growth for the bacteria, which was then used in the biological treatment of SBR. The experimental flowchart is shown in Figure 3.4-3.5.

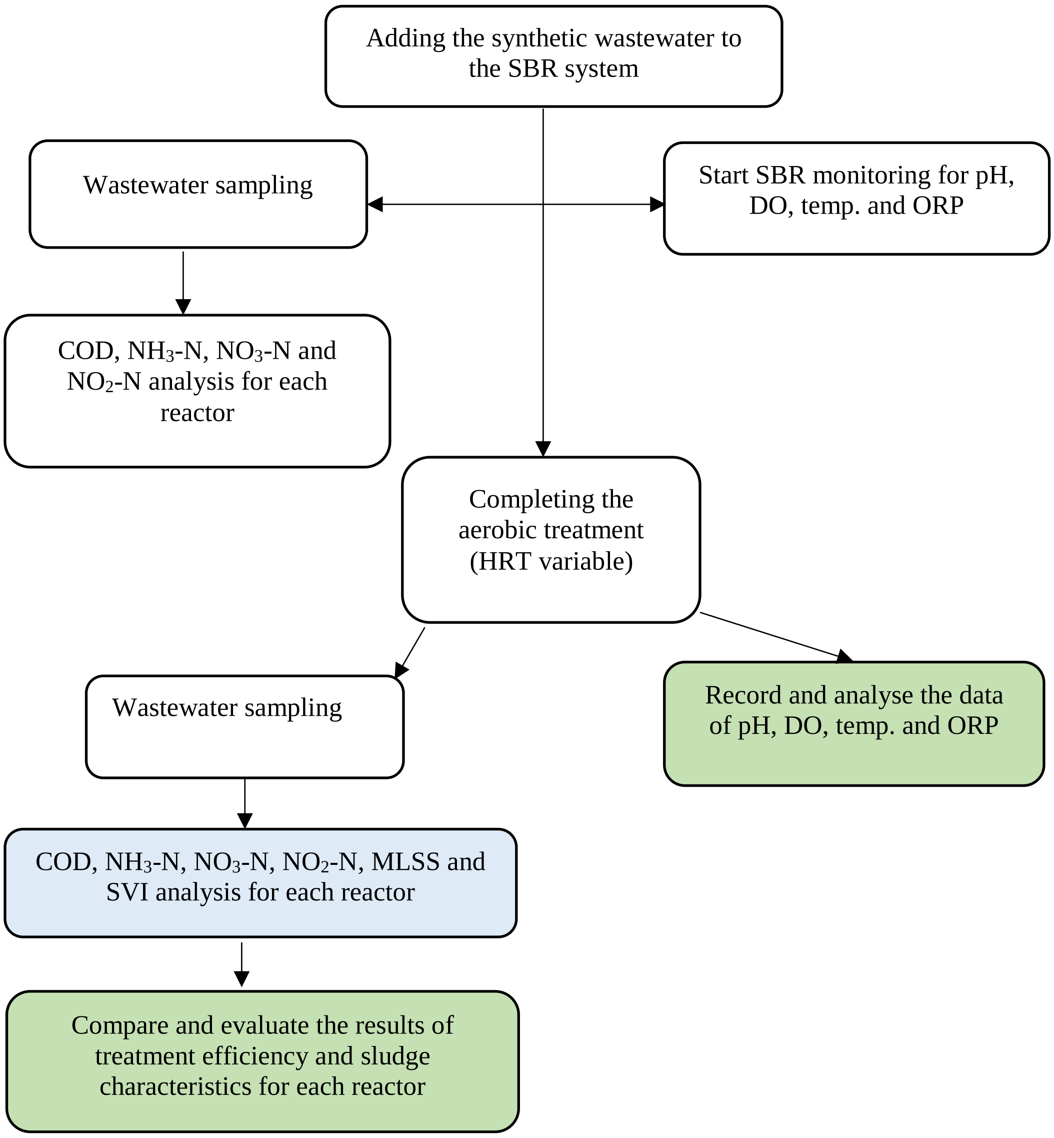



Figure 3.4: Process and experimental flow chart

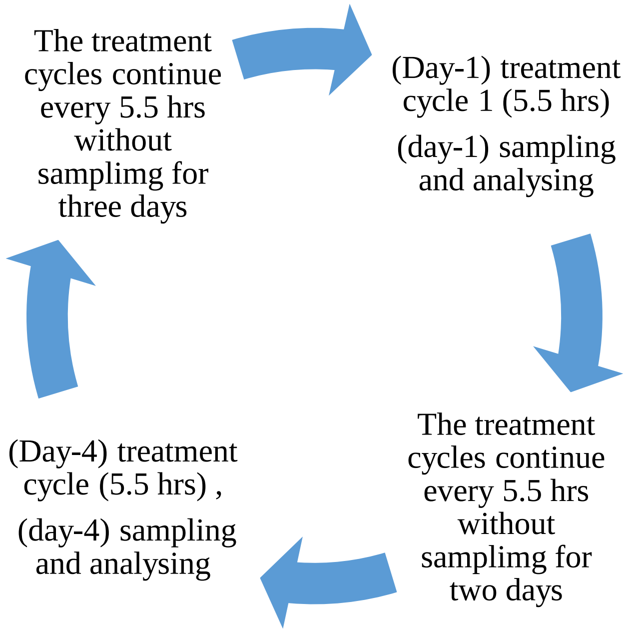



Figure 3.5: One week of operation with sampling and analysing timeline

#### 3.2.3.2 **Treatment operations**

The following flow chart (Figure 3.6) describes the methodology of sampling and testing water quality parameters. It was started by taking the sample from the reactors after adding the synthetic wastewater and analysing COD, $NH_3$-N, $NO_3$-N and $NO_2$-N. After adding the synthetic wastewater to treatment reactors, SBR starts to record the data and save it to a computer. After completing the treatment of each reactor, the sample of effluent was taken and analysed again to find the removal rates of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N and to find the SVI and MLSS to study the sludge characteristics and to evaluate the system of SBR.

To examine the removal efficiency of the two systems (NOSBR and TSSBR), two months of continuous operation and analysis will be enough to give a good idea of the systems overall efficiency. However, three months of operation will be used to examine the settling performance as the sludge settleability needs more time to show any problem with the systems (Gerardi, 2002; Jenkins et al., 2003; Leong et al., 2011).

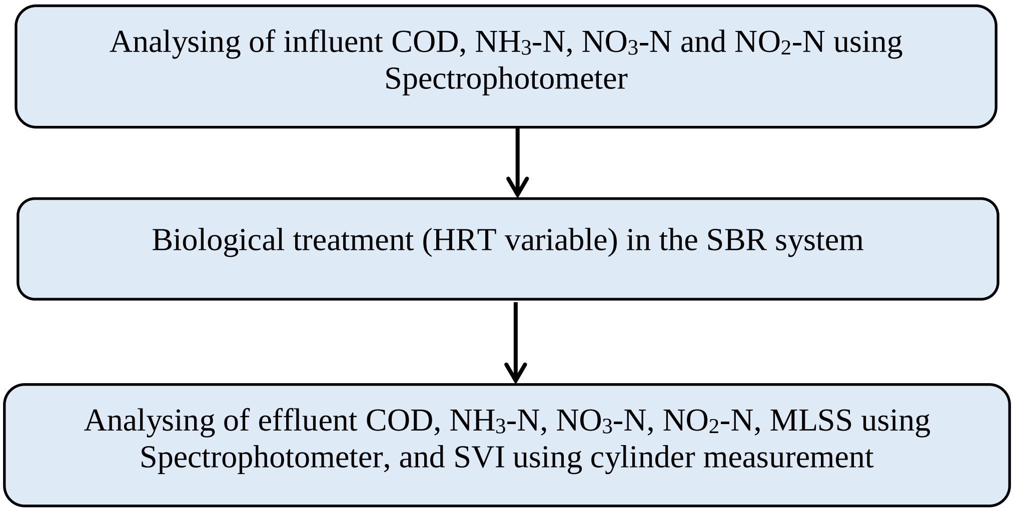



Figure 3.6: The methodology of sampling and testing water quality parameters

### 3.2.4 **Analytical methods**

In this research COD, $NH_3$-N, $NO_3$-N and $NO_2$-N were measured using HACH DR/2800 (HACH Company, 389 Loveland, Colorado USA) which is shown in Figure 3.7. Cadmium reduction method (Powder Pillows) was used to determine the $NO_3$-N concentration, while diazotization method (Powder Pillows) was used to determine the $NO_2$-N. Nessler method and Colorimetric determination were used to determine $NH_3$-N and COD respectively. All the measurements were done according to (American Public Health Association, 2012)

All the measurements were repeated three times to minimise the errors, and all the devices were calibrated weekly according to the devices manuals. The error limits were measured after each calibration, it was ± 2%, and this is acceptable according to (American Public Health Association, 2012).


Figure 3.7: HACH DR/2800 spectrophotometer

#### 3.2.4.1 **Mixed Liquor Suspended Solids**

The MLSS analysis is required to evaluate the growth of bacteria during the biological treatment of SBR. The first step is to stop the aeration in the treatment reactor of the SBR, and then wastewater with biomass were mixed properly in the reactor. The sample of 50 mL was used to analyse MLSS. Secondly, the 50 mL mixture was filtered using (0.45 µm membrane filters) as shown in Figure 3.8. The filter paper was weighed before filtration to get (X), which is the weight of filter paper before filtration. After completing the filtration, the filter paper was dried by the oven for two hours at under 150 °C. After drying, it was necessary to put the filter paper in a desiccator to remove the moisture. The filter paper was weighed again to get (Y), which is the weight of filter paper after drying as shown in Figure 3.9. Then, the MLSS was calculated to evaluate the bacterium growth. Equation (3.1) describes the MLSS calculations. Usually, (mg/l) is recommended unit for MLSS concentration.

$$MLSS\ (\frac{mg}{l}) = \frac{Y - X}{sample\ volume} \qquad (3.1)$$


Figure 3.8: Filtration device

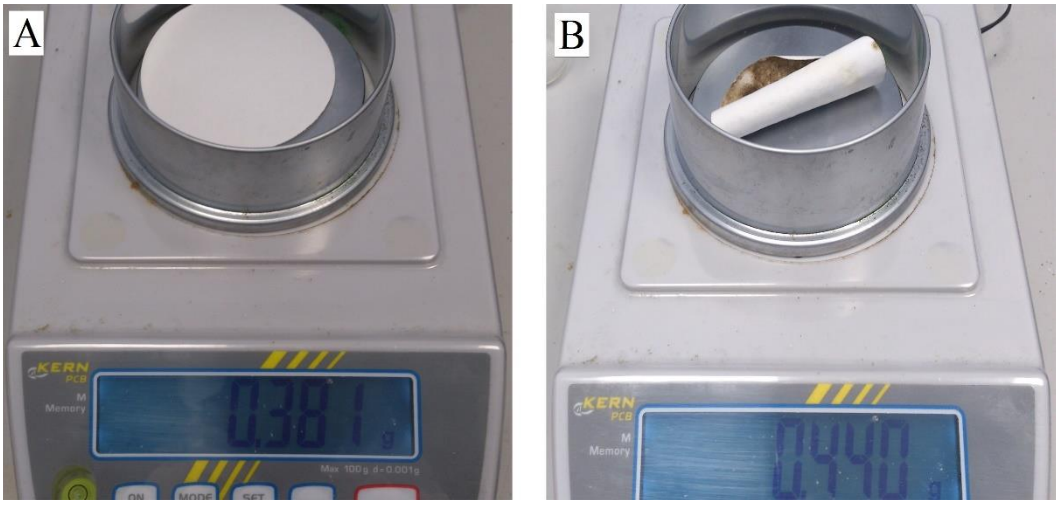



Figure 3.9: Weighting the filter paper (a) before filtration, (b) after filtration

### 3.2.4.2 **BOD**

The BOD test is used to measure waste loads to treatment plants, determine plant efficiency (in terms of BOD removal), and control plant processes. It is also used to determine the effects of discharges on receiving waters. A major disadvantage of the BOD test is the amount of time (5 days) required to obtain the results. When a measurement is made of all oxygen consuming materials in a sample, the result is termed “Total Biochemical Oxygen Demand” (TBOD), or often just simply “Biochemical Oxygen Demand” (BOD). Because the test is performed over a five-day period, it is often referred to as a “Five Day BOD”, or a $BOD_5$.

To measure the concentration of BOD, a sample is pipetted into a BOD bottle containing aerated dilution water. The DO content is determined and recorded and the bottle is incubated in the dark for five days at 20 °C. At the end of five days, the final DO content is determined and the difference between the final DO reading and the initial DO reading is calculated. The decrease in DO is corrected for sample dilution, and represents the biochemical oxygen demand of the sample.

#### 3.2.4.3 **COD**

Chemical oxygen demand (COD) is a parameter used widely to measure the pollution strength of domestic and industrial wastewaters. COD is defined as the amount of oxygen required to oxidize organic matter chemically. HACH DR 2800, USA analysing reactor model was used to measure the COD in this research. The first step in the procedure was done by taking 250 mL of the sample from the treatment reactor of SBR for analysis purposes. The reagent of the digestion solution is COD 0-1500 ppm range (high range). The second step of the procedure was to put 2 mL of wastewater sample in the reagent vial and mix properly. Then, the vial was located in the COD reactor which keeps heating for two hours under the temperature of 150°C. When the setting reaction finished, the vial was taken out of the COD reactor and cooled to room temperature for 30 minutes. When the vial cooled down, the vial was wiped out, and then the DR/2800 spectrophotometer was used to read the COD concentration. If the reading of the sample used is over the range, then the sample should be diluted.

A colorimetric determination is a method that was used to measure the COD concentration. When the spectrophotometer operates, the first step is to enter the stored program number for COD (high range), the display will show “Dial nm to 620”. When the correct wavelength is dialled in, the display will show “Zero Sample, mg/L COD HR”. Then, the COD vial adapter is placed into the cell holder, and the blank vial is prepared using 2 mL of distilled water. The vial should be cleaned before use to obtain an accurate reading by spectrophotometer. The blank is placed into the adapter with the Hash logo facing the front of the instrument, and the cover on the adapter is fixed. The spectrophotometer should be cleaned before use, and we can do that by zeroing the meters “Press Zero” the display will show “Zeroing, 0. Mg/L COD HR”.

The last step is to place the sample vial in the adapter with the Hach logo facing the front of the instrument and place the cover on the adapter. The spectrophotometer will show the reading by “Press READ”, and the results in mg/l COD will be displayed as shown in Figure 3.10.

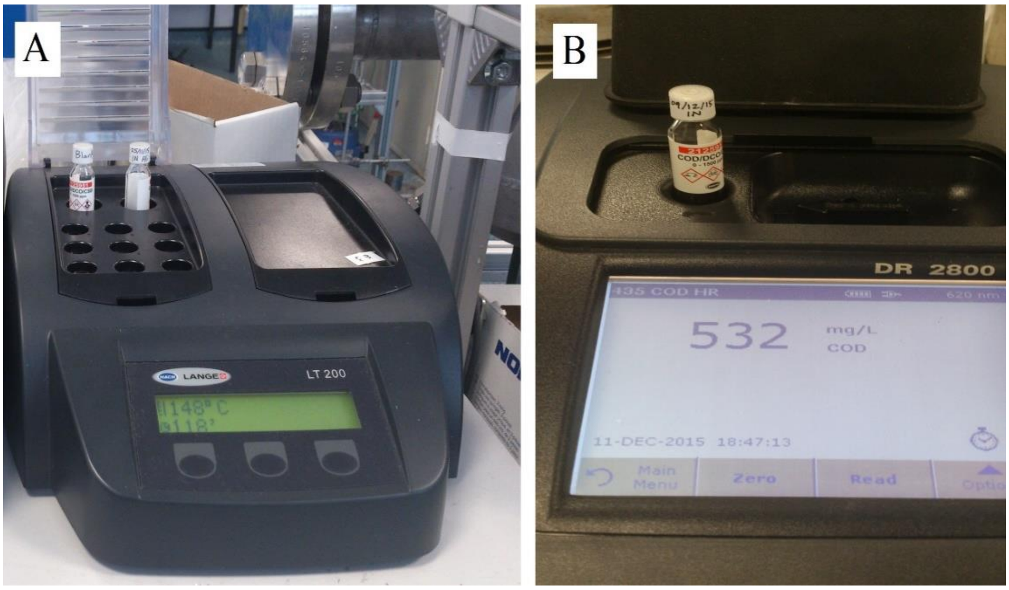



Figure 3.10: COD measurement (a) heating device, (b) spectrophotometer

Due to the time required for BOD test which is five days, industries usually use COD test instead, which requires only two hours after finding the ratio of BOD/COD for that wastewater (Ramanand Bhat et al., 2003). In this research, the BOD/COD ratio is 0.24.

#### 3.2.4.4 **$NH_3$-N**

Ammonia-nitrogen is a parameter used widely also to measure the pollution strength of industrial wastewater. A HACH DR/2800 Spectrophotometer has been used in this research to estimate the concentration of $NH_3$-N in wastewater. The first step to measure the ammonium-nitrogen is to enter the stored program number in the spectrophotometer for $NH_3$-N measurement. And then rotate the wavelength dial until the small display shows the similar number as the number clue on the small display. The second step is to prepare a blank sample by deionized water in a cylinder with a volume of 10 ml. After that, the sample of the same volume of the cylinder is prepared.

Then add three drops of Polyvinyl alcohol, and three drops from mineral stabilizer were added into samples (include the blank sample) and mix properly.

Furthermore, 1 ml Nessler reagent is added to all samples (include the blank sample) and mixed properly. After that, press SHIFT TIMER in a spectrophotometer and one minute reaction period will begin. At that time, each sample is poured into the sample cells. When the one minute reaction time finishes, the blank sample is placed in the spectrophotometer and press “ZERO” to initialize the equipment. Then, place the sample in the spectrophotometer and press “READ” to get the $NH_3$-N reading. The result will be shown in the unit of mg/l as shown in Figure 3.11. If the reading of the sample is over the range then the sample should be diluted. This method is called the Nessler method.

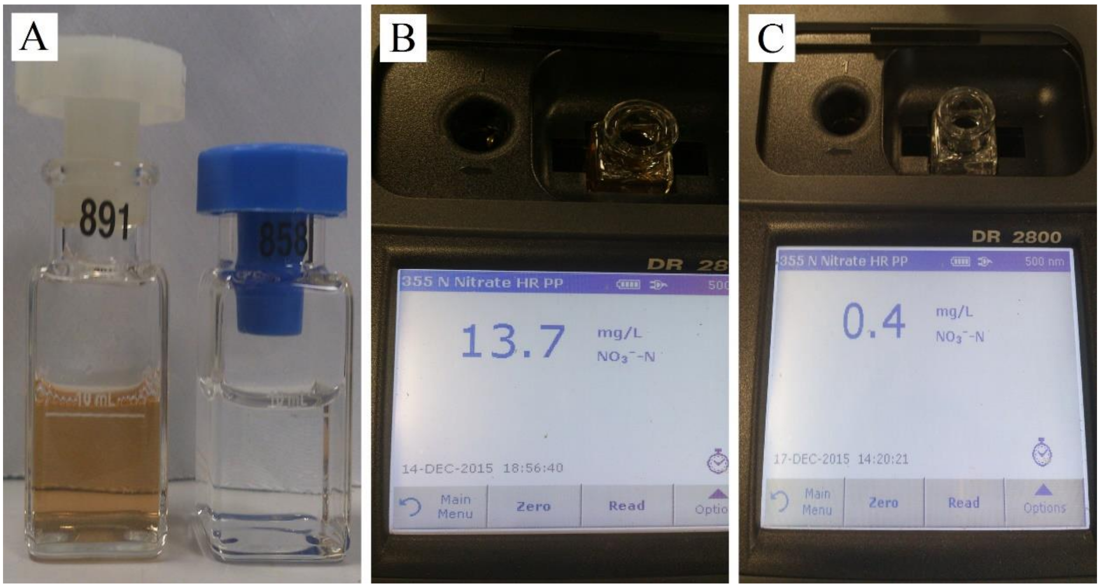



Figure 3.11: $NH_3$-N, $NO_3$-N and $NO_2$-N measurement device, (a) sample cell, (b) influent test in spectrophotometer, (c) effluent test in spectrophotometer

#### 3.2.4.5 **$NO_3$-N**

Cadmium reduction method (Powder Pillows) was used to determine the $NO_3$-N concentration. The first step to measure the concentration of nitrate (high range) is to enter the stored program number in the spectrophotometer for $NO_3$-N estimation. Then rotate the wavelength dial to the same as the prompted number shown in the small display and then “ENTER” is pressed.

After filling the sample cell with 10 ml sample and adding the content of nitrate reagent high range powder pillow to the cell, the cell is shaken vigorously for 1 minute, and then it is left to react for 5 minutes. At that time, another sample cell is filled with blank sample. When the 5 minutes reaction time finishes, the blank sample is used for initializing the equipment, and then the sample is placed in the spectrophotometer and “READ” is pressed to get $NO_3$-N reading. The result will show in the unit of mg/l as shown in Figure 3.11. If the reading of the sample is over range then the sample should be diluted.

#### 3.2.4.6 **$NO_2$-N**

Diazotization method (Powder Pillows) was used to determine the $NO_2$-N. The first step is to measure the concentration of nitrite (low range) and then to enter the stored program number in the spectrophotometer for $NO_2$-N estimation. Then, the wavelength dial is rotated to the same as the prompt shown in the small display and press “ENTER”.

After that, a sample cell is filled with 10 ml sample, and the content of nitrite reagent low range powder pillow is added to the cell. Then, the cell is shaken vigorously for 1 minute and then “SHIFT TIMER” is pressed. A 20 minutes reaction will begin. At that time, another sample cell is filled with blank sample. When the 20 minutes reaction time finishes, use the blank sample for initializing the equipment and then place the sample in the spectrophotometer and press "READ" to get $NO_2$-N reading. The result will show in the unit of mg/l as shown in Figure 3.11. If the reading of the sample is over the range, then the sample should be diluted.

#### 3.2.4.7 **Settled sludge volume**

Settled sludge volume (SSV) of a biological suspension is very useful in routine monitoring of the activated sludge process. A 30-minutes settled sludge volume has been used to monitor the return activated sludge (RAS) and when is required to remove some of the sludge from the system. In addition, 30-minutes settled sludge volume is used to find the sludge volume index.

SSV is measured using 1L cylinder measurement and 1L of sludge sample. The sludge sample should be taken from the aeration basin after good mixing, and placed in the 1L cylinder measurement. Then, the volume occupied by suspension should be determined at measured time intervals, e.g., 5, 10, 15, 20, 25 and 30 minutes (American Public Health Association, 2012).

#### 3.2.4.8 **Sludge volume index**

Sludge volume index (SVI) is the volume in (ml) occupied by 1 gram of a suspension after 30 minutes of settling. Sludge volume index is used to monitor the settling sludge performance of the activated sludge process and other biological suspensions. After performing the settled sludge volume mentioned above, the SVI can be measured as shown in Equation (3.2) (American Public Health Association, 2012). SVI measurement is shown in Figure 3.12.

$$SVI\ (\frac{ml}{g}) = \frac{settled\ sludge\ volume\ \left(\frac{ml}{l}\right) x1000}{suspended\ solids\ (\frac{mg}{l})} \tag{3.2}$$

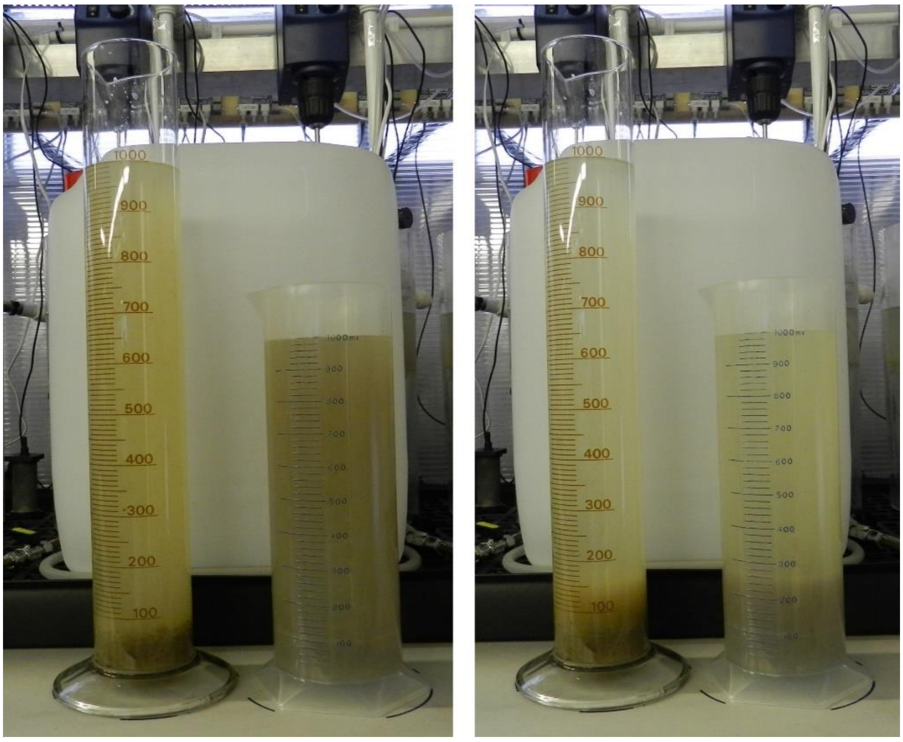
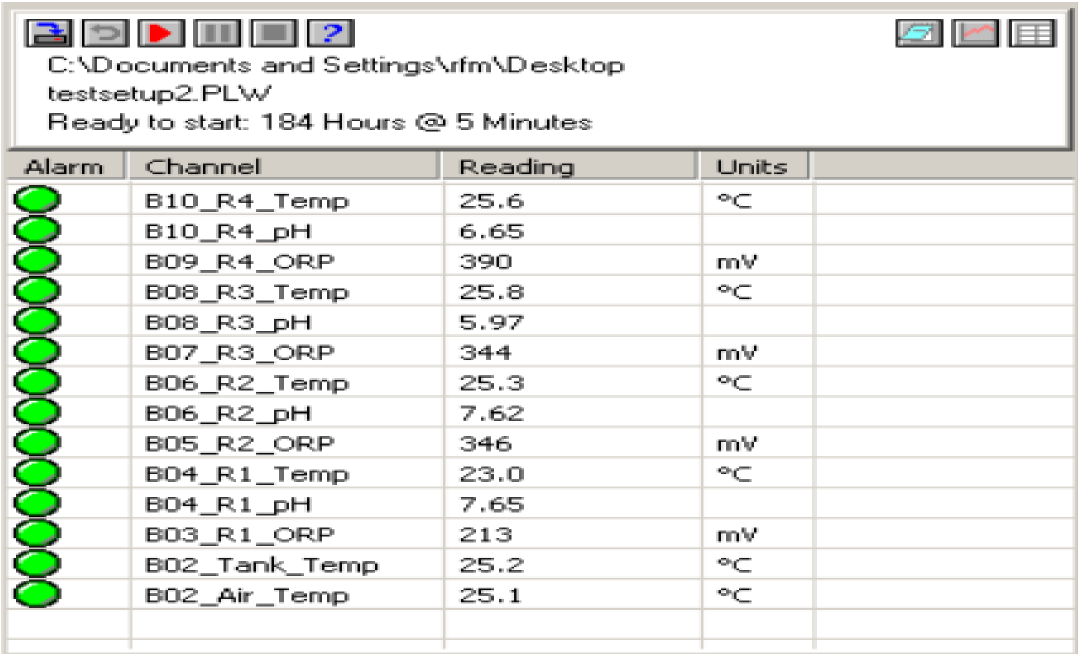



Figure 3.12: Sludge volume index test

### 3.2.5 **Online monitoring system for SBR**

The main challenge in the management of the sequencing batch reactor, for the biological treatment of industrial wastewater, is to ensure stable treatment efficiency under highly variable influent quality and quantity. To help SBR operators to cope with this challenge, online programming is fundamental, since it allows describing both influent variability and process efficiency. So, the process monitoring of an SBR is necessary to ensure the proper operation. Each SBR that is used in this research contains four digital meters for pH, DO, temperature and ORP profile measurement linked to a personal computer (PC) by cable to transfer the data automatically. The digital meters are contacted with sensors (probe) in treatment reactors of the SBR. The probes and the software were provided by Pico Technology, UK.

The program was designed to present and record the data automatically by computer. The program monitors the changing of pH, DO, temperature and ORP profiles with time as shown in Figure 3.13.




| Alarm | Channel | Reading | Units |
|---|---|---|---|
| | B10_R4_Temp | 25.6 | °C |
| | B10_R4_pH | 6.65 | |
| | B09_R4_ORP | 390 | mV |
| | B08_R3_Temp | 25.8 | °C |
| | B08_R3_pH | 5.97 | |
| | B07_R3_ORP | 344 | mV |
| | B06_R2_Temp | 25.3 | °C |
| | B06_R2_pH | 7.62 | |
| | B05_R2_ORP | 346 | mV |
| | B04_R1_Temp | 23.0 | °C |
| | B04_R1_pH | 7.65 | |
| | B03_R1_ORP | 213 | mV |
| | B02_Tank_Temp | 25.2 | °C |
| | B02_Air_Temp | 25.1 | °C |

Figure 3.13: The pH, DO, temperature and ORP profiles is recorded with time

## 3.3 Morphological study and image analysis

Activated sludge samples were collected from each treatment reactor (TSSBR and NOSBR) and analysed on the same day to examine the sludge settleability for both systems.

Sludge settling is a critical issue in most treatment plants as it can increase the time needed for treatment which increases operation costs. The sludge volume index is the most common indicator of sludge settleability (Metcalf and Eddy, 2014). SVI has been used widely to test sludge settleability in both laboratory scale and pilot plant scale studies (Trelles et al., 2017). The settleability of activated sludge systems can also be monitored and controlled through microscopic observation (Mesquita et al., 2011; Tomperi et al., 2017). Quantitative image analysis is a promising technique which has been used to study different problems in activated sludge systems (Grijspeerdt and Verstraete, 1997; Jassby et al., 2014; Mesquita et al., 2011; Wagner et al., 2015). In this research, $SVI_{30}$ was used to determine settling performance along with a quantitative study for sludge samples which targeted the filamentous bacteria as this is considered one of the main reasons for sludge settling problems as mentioned earlier.

A light microscope (AX10, Zeiss, Germany) with a colour video camera (PixeLINK, Canada), which is shown in Figure 3.14, was used to examine the morphological characteristics of the sludge by capturing images and analysing them via image processing software. Over the whole period of the study, samples were taken from both treatment reactors every other day to record differences in filamentous growth and the diversity of sludge characteristics between the reactors to relate this to sludge settleability. Pictures were taken at 100x magnification. Two microscope slides were used for each sample, and for each slide, 10µL of the sample was poured onto the slide using a micropipette (Mesquita et al., 2011). A total of 80 images were captured for each sample (40 images per slide) to avoid bias. A quantitative study of the captured images was conducted by studying the ratio of total filament length per MLSS value (TL/MLSS), and the ratio of total filament length per sample volume (TL/ Vol). This was achieved using the same method as Mesquita et al. (2010). Image acquisition, background pre-treatment, aggregate segmentation, filamentous segmentation and debris elimination were carried out as shown in Figure 3.15, using MATLAB 9 (The Mathworks, Natick, USA), following Mesquita et al. (2010) procedure.

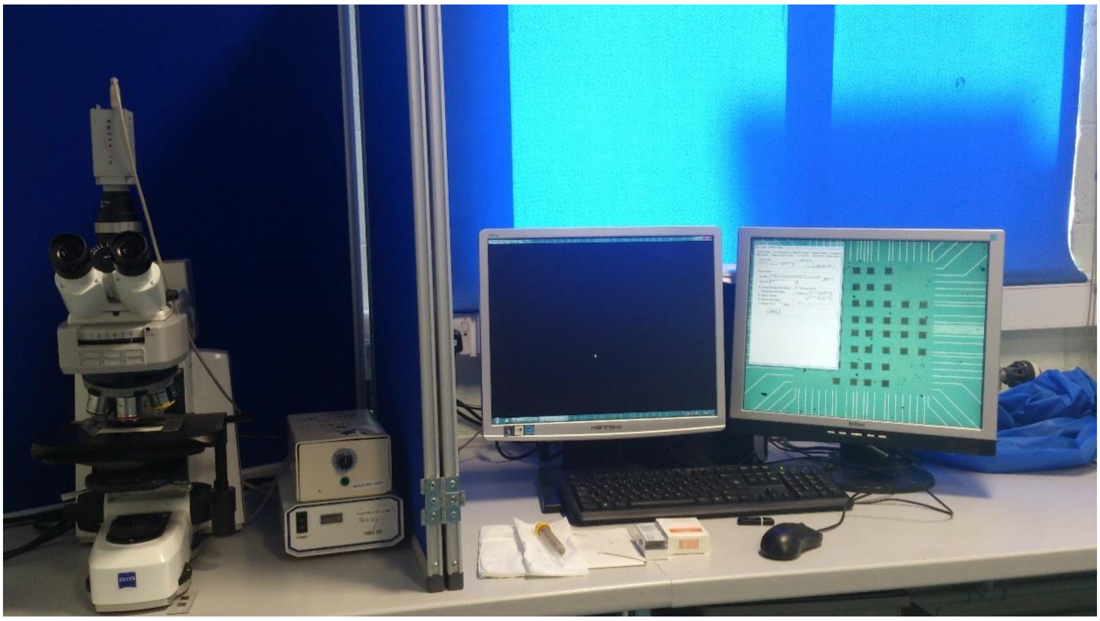


Figure 3.14: A light microscope with a colour video camera

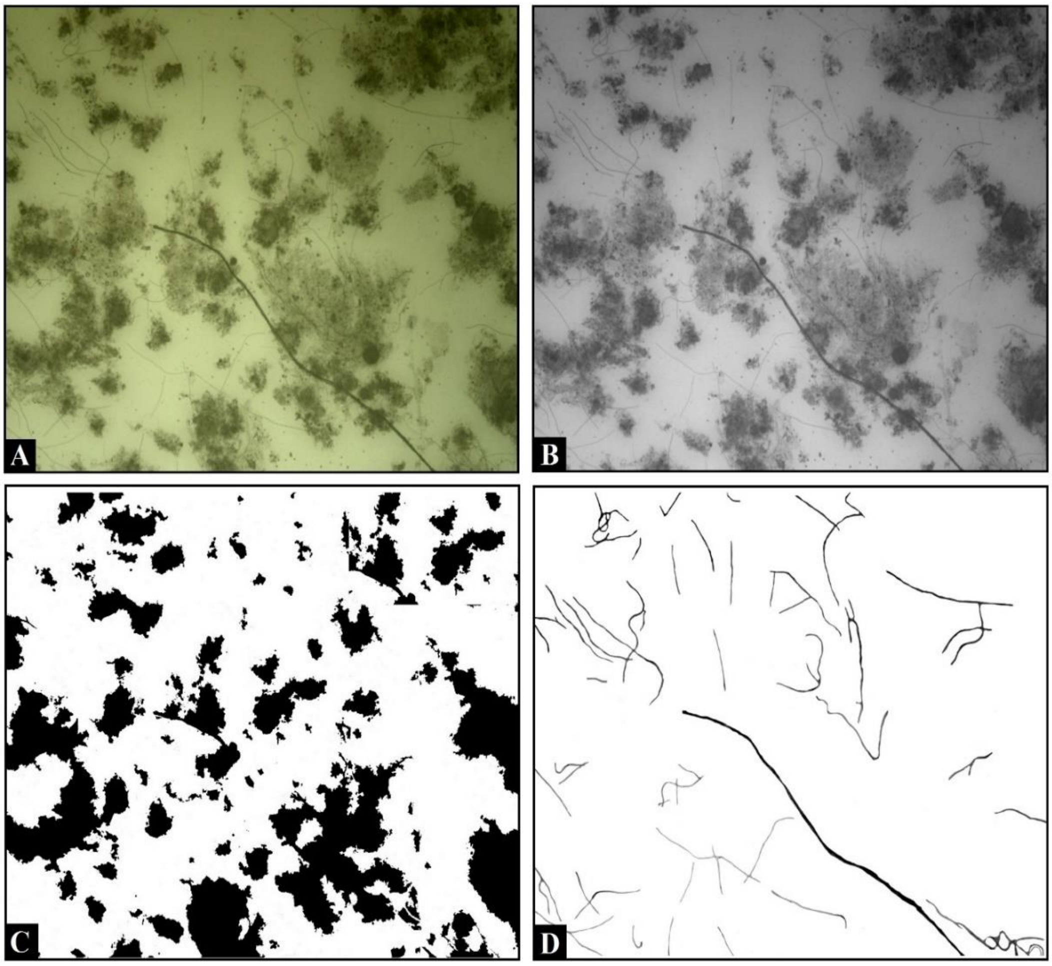



Figure 3.15: Schematic representation of image processing program. (a) Image acquisition, (b) background pre-treatment, (c) aggregate segmentation, (d) filamentous segmentation and debris elimination.
Source (Alattabi et al., 2017c)

### 3.4 Scanning electron microscopy

In addition to the microscopic study of the sludge, scanning electron microscopy (SEM) analysis was conducted to establish the differences between NOSBR and TSSBR in terms of sludge characteristics and settleability performance by targeting the filamentous bacteria in the sludge samples and relating this to the morphological study and SVI values. If an abundance of filamentous bacteria is present, the sludge is settling slowly and vice versa. SEM analysis was carried out using INCA x-act, OXFORD Instruments, UK. Kalab et al. (2008) method was followed to prepare the samples for SEM analysis, which is shown below.

The sludge samples were taken from both reactors (NOSBR and TSSBR) and fixed with 2% glutaraldehyde and stored for 24 hours at 4 °C. For a specimen to be analysed by SEM, the specimens are then washed in alcohol dehydration by using different concentrations of sequentially 30%, 50%, 70%, 80% and 90% respectively for 30 min, and after that 100% of three times each 30 minutes. The next sample is transferred into the specimen container and placed in a critical stage dryer for 30 min and placed on the plate using double-sided tape coated with gold using a sputter coater before SEM pictures were taken. The SEM device is shown in Figure 3.16.

Figure 3.16: SEM device

## 3.5 Statistical Analysis

Statistical analyses have been performed to assess the performance of the studied reactors, TSSBR and NOSBR, regarding SVI and the removal of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N. Three key parameters were investigated using SPSS; the standard deviation, outliers and the normality (according to Kolmogorov-Smirnov test) of the obtained results. The standard deviation describes the amount of variation in the parameter under investigation; the smaller the standard deviation, the better the consistency and quality of the treatment process (Armstrong et al., 2017; Hashim et al., 2017a; Wachs, 2009). The presence of outliers, which could be defined as extreme observations, indicates a poor and unstable performance, while the normality of the obtained results enhances the ability to model treatment performance (Hashim et al., 2017b;c; Jafer et al., 2016).

## 3.6 Chapter summary

This chapter showed the design description of the two-stage settling SBR that was used in this research project to enhance the solids settling performance and showed as well the cost consideration for proposing this system to the industry. In addition, the methodology that was used in this research project including (the activated sludge source, synthetic wastewater, lab-scale SBR description and treatment operation) has been illustrated in this chapter. Also, the analytical methods of (MLSS, COD, $NH_3$-N, $NO_3$-N, $NO_2$-N, SSV and SVI) were described in this chapter. Moreover, the online monitoring system (pH, DO, ORP and temperature) was described in this chapter.

Morphological study of the sludge is essential to assess the settling performance of the solids and support the SVI test that measures the settling performance. Therefore, morphological study and image analysis along with the scanning electron microscopy were conducted in this research and described in this chapter.

Finally, to evaluate the results and measure the significance of the results acquired from both reactors, a statistical analysis using SPSS was conducted in this research, and the statistical analysis methods for the obtained results were described in this chapter. The performance of both systems (TSSBR and NOSBR) will be illustrated in the next chapter (Chapter 4)

CHAPTER 4

# Treatment efficiency and sludge settleability of NOSBR and TSSBR

## 4.1 Treatment efficiency of TSSBR and NOSBR

In this research, the SBR system was operated for more than 450 days continually at ambient temperature to study all the research objectives. The performance of the TSSBR was compared with that of a normal operating sequencing batch reactor, operating with the same cycle time for three months. The parameters of pH, ORP, temperature and DO have been monitored online during the daily experiments. The results of experiments will be explained through graphs for influent, effluent and the treatment efficiency in the following sections.

One reactor was used for the normal operation sequencing batch reactor, and another reactor was used for the two-stage settling sequencing batch reactor with the same cycle times (5.5 h). The operation cycles for the NOSBR and the TSSBR are shown in Table 4-1. All the results of this study are within the UK regulations of wastewater discharge, which listed in Table 2-2.

Table 4-1: NOSBR and TSSBR operation cycles
Source (Alattabi et al., 2017c)

| NOSBR | Anoxic Fill | React | Settle | | | Draw & Idle |
|---|---|---|---|---|---|---|
| Time (min) | 15 | 240 | 60 | | | 15 |
| TSSBR | Anoxic Fill | React | Settle I | Mixing | Settle II | Draw & Idle |
| Time (min) | 15 | 240 | 15 | 15 | 30 | 15 |

### 4.1.1 Acclimatisation stage of SBR

Over the whole period of study, the sludge age was kept at between 12-18 days (Ekama, 2010). The acclimatisation stage for the bacterial culture in the treatment reactors lasted for 20-30 days, after this period the system has reached its steady state in which the removal efficiency acceded 80% (Ekama, 2010).

### 4.1.2 Treatment performance of TSSBR and NOSBR

#### 4.1.2.1 COD removal efficiency

The efficiency of the removal of COD for the NOSBR and TSSBR is shown in Figure 4.1. The average efficiency for the removal of COD in the NOSBR and TSSBR was 93.7% and 93.1%, respectively, the average effluent 54.83 mg/l and 54.7 mg/l, respectively. The similarities in efficiency for both reactors could be due to the same reaction time and operating conditions over both. Some outliers were noticed on the graph; the reason for this could be the addition of new sludge every 20 days that could affect the treatment performance of the system.

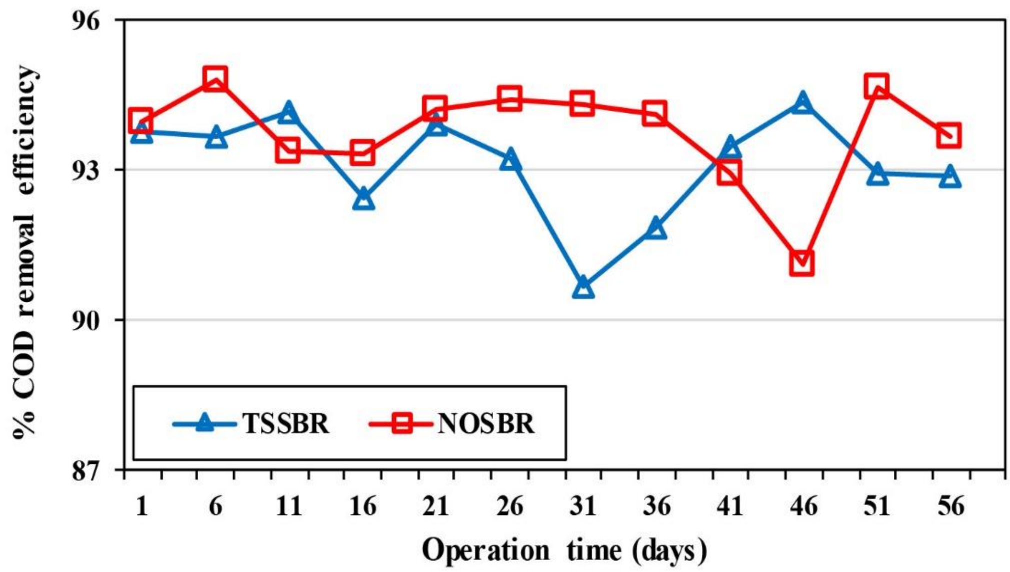



Figure 4.1: COD removal efficiency of NOSBR and TSSBR
Source (Alattabi et al., 2017c)

#### 4.1.2.2 $NH_3$-N removal efficiency

The efficiency of the removal of $NH_3$-N for the NOSBR and TSSBR is shown in Figure 4.2. The average efficiency of removal of $NH_3$-N for the NOSBR was 76.6% with an average effluent of 1.87 mg/l. While the average efficiency of the removal of $NH_3$-N for the TSSBR was 89.2% with an average effluent of 0.85 mg/l.

Nitrosomonas and Nitrobacter bacteria generally grow as single cells but may be held together. They are extremely sensitive to any changes in their environment and die off because acute toxicity is common. The removal rates change with bacterium growth and activities in the treatment tank. Nitrosomonas is a type of autotrophic bacterium which has the ability to oxidize the ammonia to nitrite based on the overall reaction as shown in Equation (4.1):

$$NH_3 + \frac{3}{2} O_2 \longrightarrow NO_2^- + H^+ + H_2O \quad (4.1)$$

The oxidation of $NH_3$ to $NO_2^-$ is a process in which energy can be generated. Thus, bacteria use this energy to assimilate carbon dioxide. Again, few outliers were noticed on the graph; the reason for this could be the addition of new sludge every 20 days that could affect the treatment performance of the system.

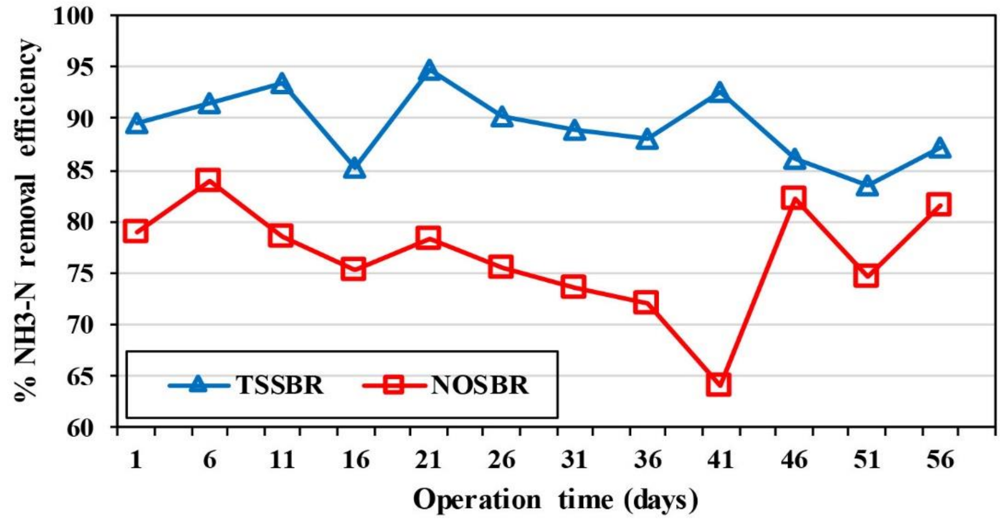



Figure 4.2: $NH_3$-N removal efficiency of NOSBR and TSSBR
Source (Alattabi et al., 2017c)

#### 4.1.2.3 **$NO_3$-N removal efficiency**

The efficiency of the removal of $NO_3$-N for the NOSBR and TSSBR is shown in Figure 4.3. The average efficiency of removal of $NO_3$-N for the NOSBR was 86.4% with an average effluent of 2.41 mg/l. While the average efficiency of the removal of $NO_3$-N for the TSSBR was 95.2% with an average effluent of 0.81mg/l. Same reason above led to show few outliers on the graph.

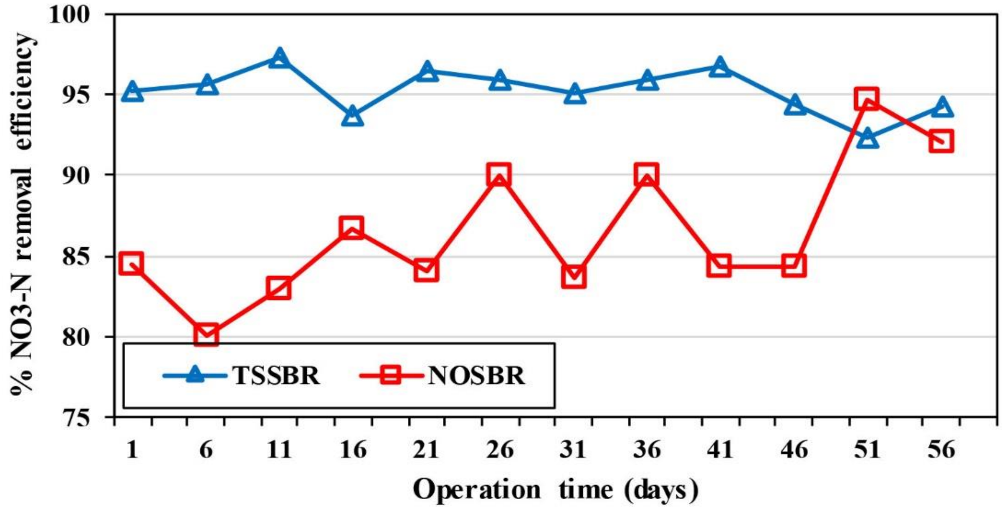



Figure 4.3: $NO_3$-N removal efficiency of NOSBR and TSSBR
Source (Alattabi et al., 2017c)

#### 4.1.2.4 **$NO_2$-N removal efficiency**

The efficiency of the removal of $NO_2$-N for the NOSBR and TSSBR is shown in Figure 4.4. The average efficiency of removal of $NO_2$-N for the NOSBR was 87.3% with an average effluent of 2.23 mg/l. While the average efficiency of the removal of $NO_2$-N for the TSSBR was 96% with an average effluent of 0.75 mg/l.

The reason for improving the removal efficiency of $NH_3$-N, $NO_3$-N and $NO_2$-N in the TSSBR over the NOSBR could be the enhancement of the nitrogen cycle by offering the anoxic stage between the two settling stages in the TSSBR. During the anoxic fill, ammonia can be decreased by half (She et al., 2016), and denitrification might be occurring due to low DO concentrations and the presence of a carbon source. Ammonium was oxidized completely during the aeration stage while the remaining nitrate and nitrite were removed during the second anoxic stage in the TSSBR. This is the reason why the nitrogen compounds were removed more effectively in TSSBR in comparison to NOSBR. These results substantiate the work of (Chen et al., 2013) who studied a step-feeding SBR and achieved high nitrogen removal rates during two aeration phases. Chen et al. (2013) also stated that the anoxic condition during the feeding stage could result in high rate of denitrification which, in turn, leads to high nitrogen removal efficiency. The addition of new sludge every 20 days could be the reason for the outliers on the graph.

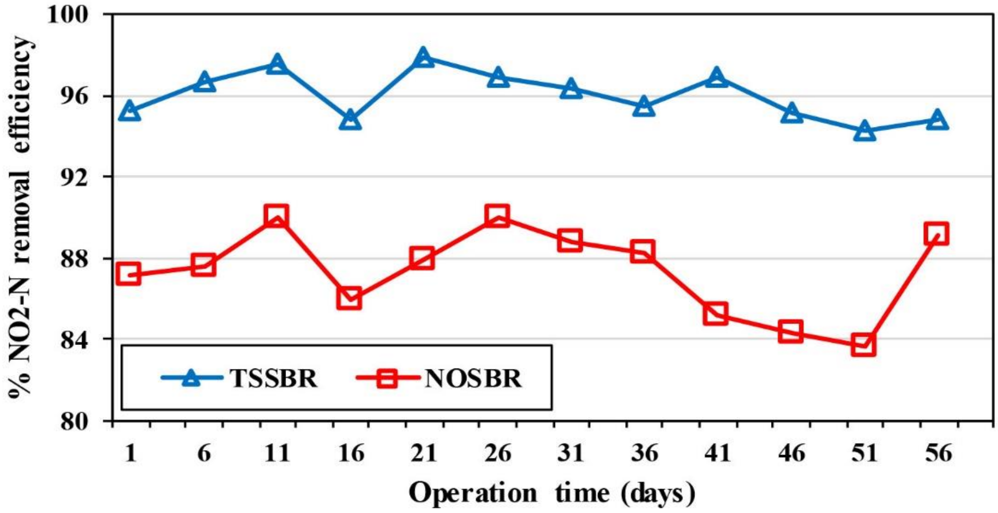



Figure 4.4: $NO_2$-N removal efficiency of NOSBR and TSSBR
Source (Alattabi et al., 2017c)

#### 4.1.2.5 **Mixed liquor suspended solid**

Mixed liquor suspended solids was studied for the treatment reactors of TSSBR and NOSBR twice a week for the whole period of study at ambient temperature. Ambient temperature was used in this study rather than a stable temperature to measure the system performance during a various period of the year at the UK weather. Besides, in the real wastewater treatment plant, ambient temperature is used because it is difficult and costly to control the wastewater temperature. Over the three-month operation time, the sludge age was kept at between 12-18 days; the MLSS concentration was ranged between 2500 mg/l and 3500 mg/l for both reactors (NOSBR and TSSBR) as can be seen in Figure 4.5.

The growth of biomass (MLSS concentration) in this research was unstable as shown in Figure 4.5. It is affected by chemical and physical parameters such as temperature, pH, oxygen level, moisture, nutrient content and minerals. The temperature is one of the important factors that have an effect on the growth of microbial. The growth can occur at temperatures below freezing or up to more than 100°C, based on the ideal temperature for growth. The recent studies have shown that the suitable pH for microbial growth is around 7 and the presence of oxygen is necessary for nitrifiers to oxidize the ammonia to nitrate (Bitton, 2005).

Slater et al. (2005) operated the membrane bioreactor (MBR) with MLSS of 12,000 mg/L, while a typical SBR operated with MLSS of 3,000 mg/L. This difference in biomass concentration leads to much smaller process basins for MBR technology and results in the MBR system having an overall plant footprint 50-70% smaller than an SBR system. Because it relies on phase separation, the SBR cannot operate at elevated biomass concentrations, as the sludge loses its ability to settle into distinct layers once the MLSS gets above 6,000-8,000 mg/L.

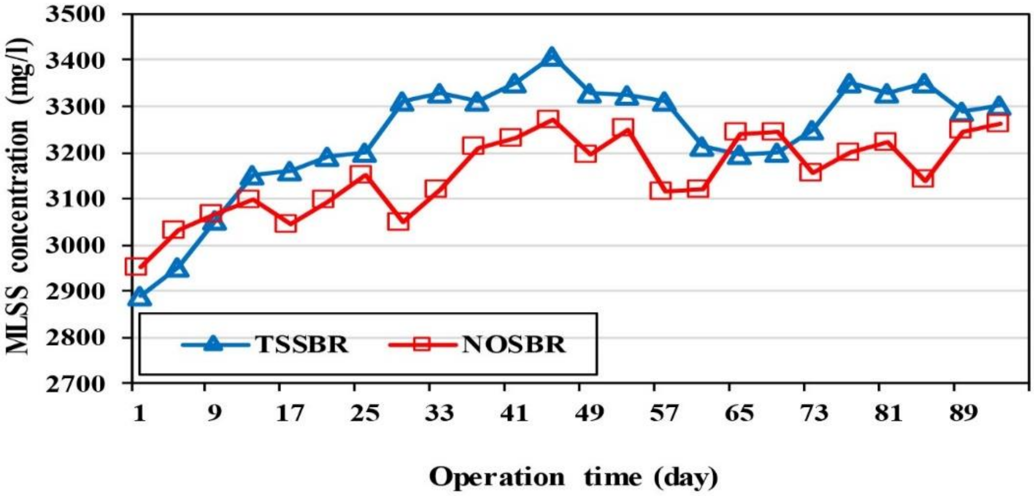



Figure 4.5: MLSS of NOSBR and TSSBR
Source (Alattabi et al., 2017c)

## 4.2 Sludge settleability of TSSBR and NOSBR

Sludge settling is a critical issue in most treatment plants as it can increase the time needed for treatment which increases operating costs. The sludge volume index is the most common indicator of sludge settleability (Metcalf and Eddy, 2014). The settleability of activated sludge systems can also be monitored and controlled through microscopic observation (Mesquita et al., 2011). Quantitative image analysis is a promising technique which has been used to study different problems in activated sludge systems (Grijspeerdt and Verstraete, 1997; Jassby et al., 2014; Mesquita et al., 2011; Wagner et al., 2015)

### 4.2.1 Settled sludge volume and sludge volume index

In this research, SSV and SVI were used to determine settling performance along with a quantitative study for sludge samples by targeting the filamentous bacteria because it is considered one of the main reasons for sludge settling problems as mentioned earlier. As shown in Figure 4.6 and Figure 4.7, the settling ability of the TSSBR is better than the NOSBR. The average SVI for TSSBR and NOSBR was 31.17 ml/g and 42.04 ml/g, respectively.

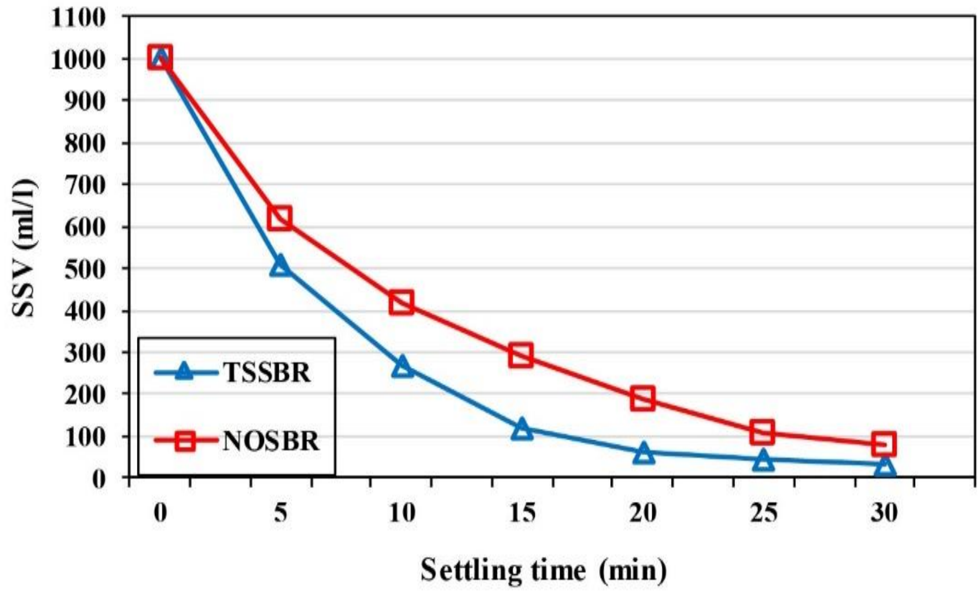



Figure 4.6: Settled sludge volume for NOSBR and TSSBR
Source (Alattabi et al., 2017c)

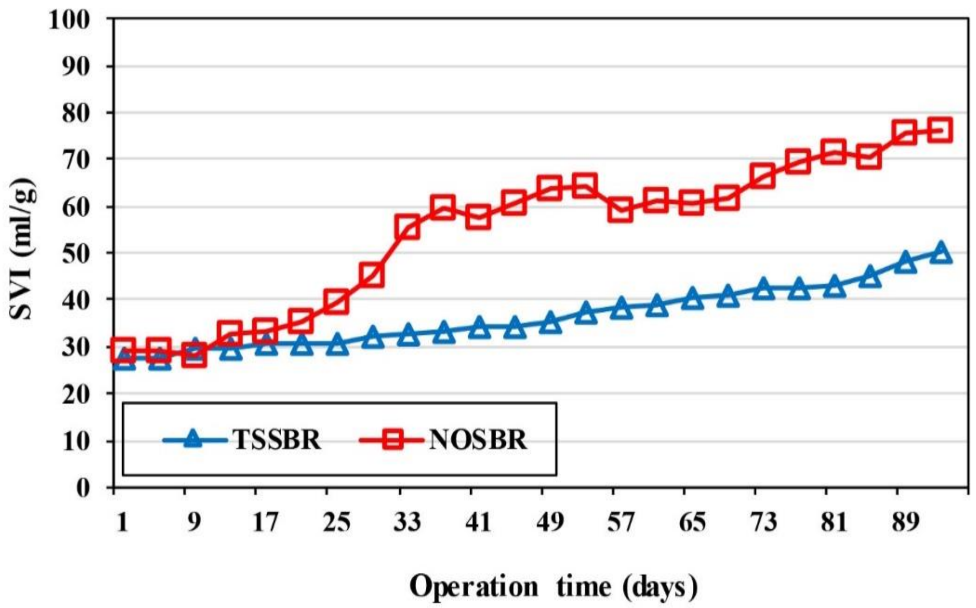



Figure 4.7: Sludge volume index for NOSBR and TSSBR
Source (Alattabi et al., 2017c)

### 4.2.2 Morphological study and image analysis

The quantitative microscopic study of filamentous growth which is shown in Figure 4.8 and Figure 4.9 reported the same results mentioned in the previous section. The average TL/MLSS for TSSBR and NOSBR were 1475.33 mm/mg and 1594.34 mm/mg, respectively, and the average TL/Vol were 139.70 mm/µl and 221.79 mm/µl, for TSSBR and NOSBR respectively. The individual aggregate area (Area) was determined as the pixel sum of each aggregate projected surface calibrated to metric units by a calibration factor $F_{Cal}$ (µm pixel$^{-1}$) determined by the use of a micrometer slide. The filaments individual length (FL) was determined according to (Walsby and Avery, 1996), (equation 4.2) with $N_{Thn}$ as the pixel sum of each thinned filament, $N_{int}$ as the number of filament intersections, and factor 1.1222 used to average the different measuring angles within the image. Once again the obtained values were calibrated to metric units by the use of the $F_{Cal}$ (µm pixel$^{-1}$) calibration factor:

$$FL = ( N_{Thn+} N_{int}) \times 1.1222 \times F_{Cal} \quad (4.2)$$

Then FL was divided by the MLSS to obtain the TL/MLSS, and FL was divided by the sample volume to obtain the TL/Vol.

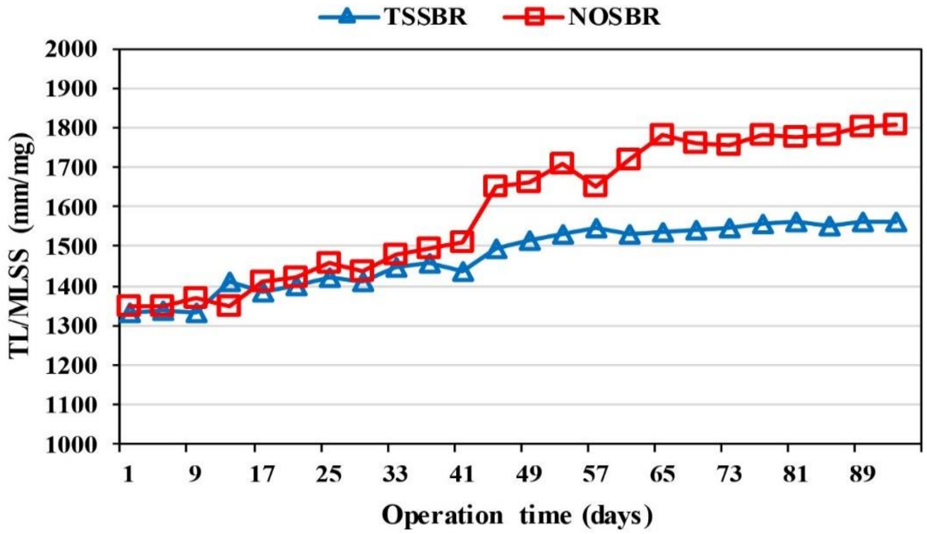



Figure 4.8: filament total length per MLSS for NOSBR and TSSBR
Source (Alattabi et al., 2017c)

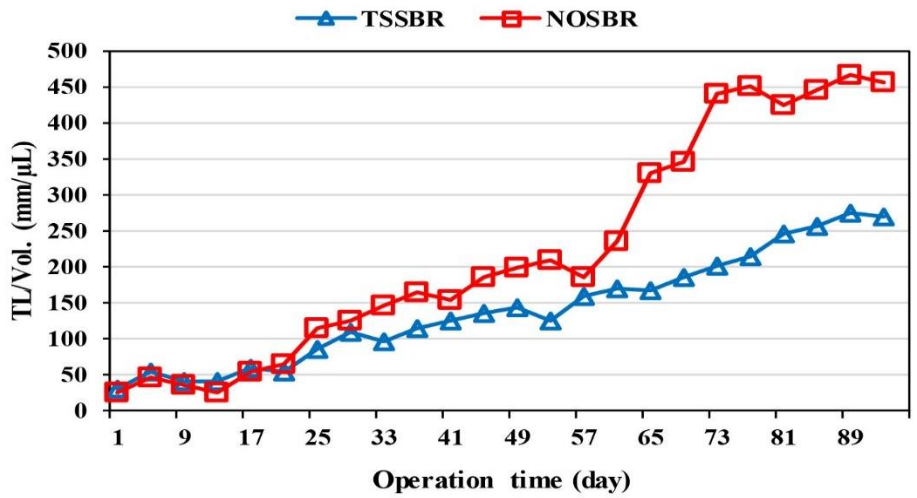



Figure 4.9: filament total length per the sample volume for NOSBR and TSSBR
Source (Alattabi et al., 2017c)

By plotting the results of TL/MLSS and TL/Vol with SVI results, a highly significant relationship was found ($R^2 = 0.93$) as shown in Figures 4.10-4.14, which means that the total length of filamentous bacteria affects sludge settleability.

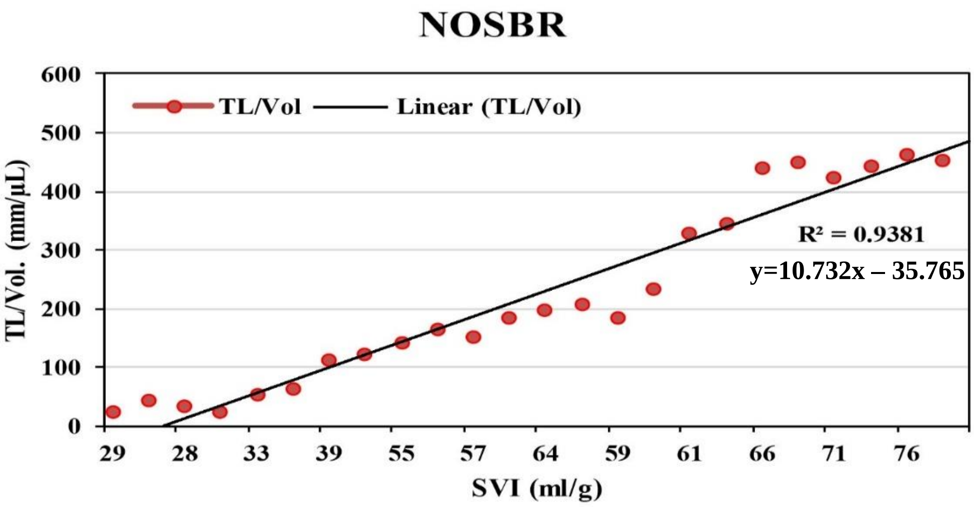



Figure 4.10: filament total length per the sample volume vs. SVI for NOSBR
Source (Alattabi et al., 2017c)

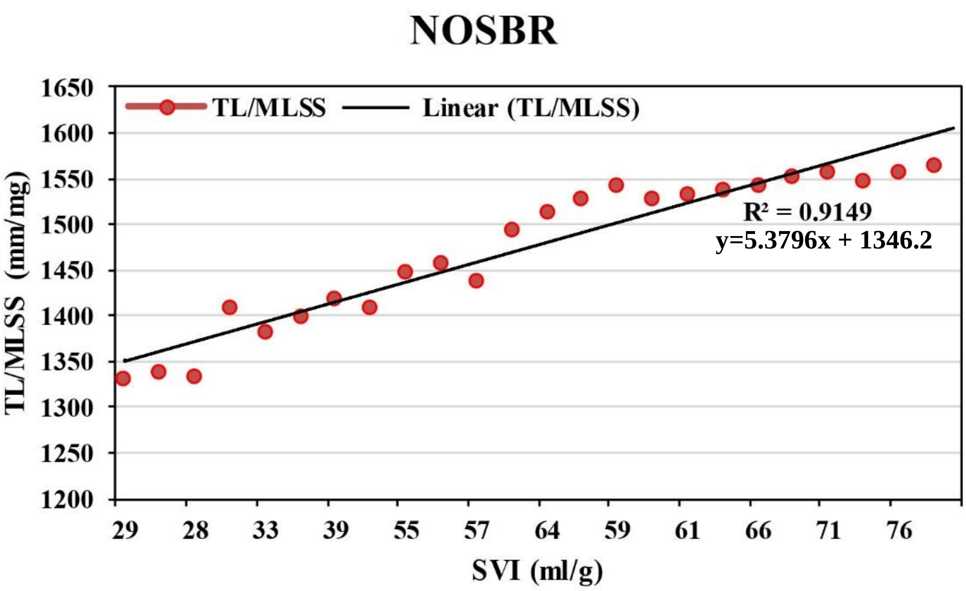



Figure 4.11: filament total length per MLSS vs. SVI for NOSBR
Source (Alattabi et al., 2017c)

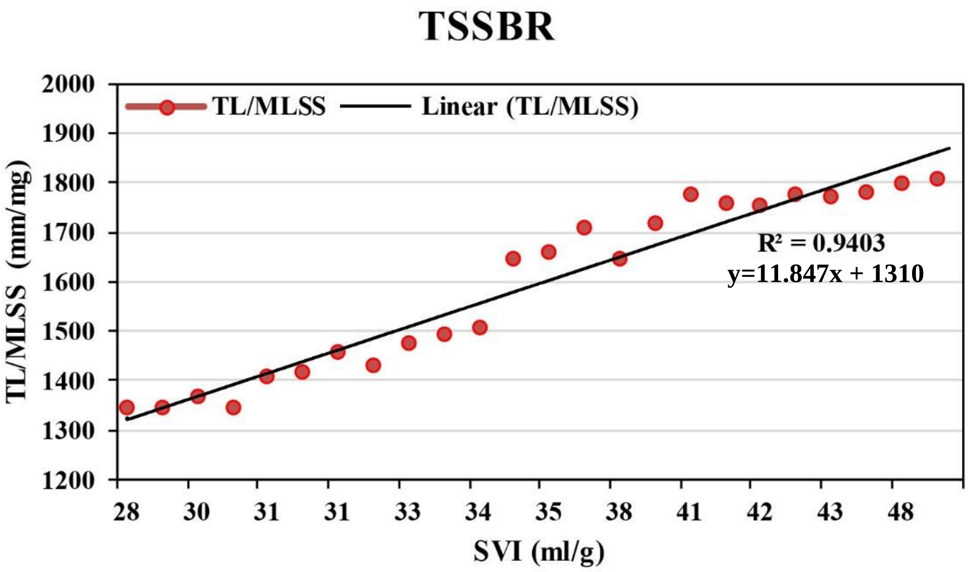



Figure 4.12: filament total length per MLSS vs. SVI for TSSBR
Source (Alattabi et al., 2017c)

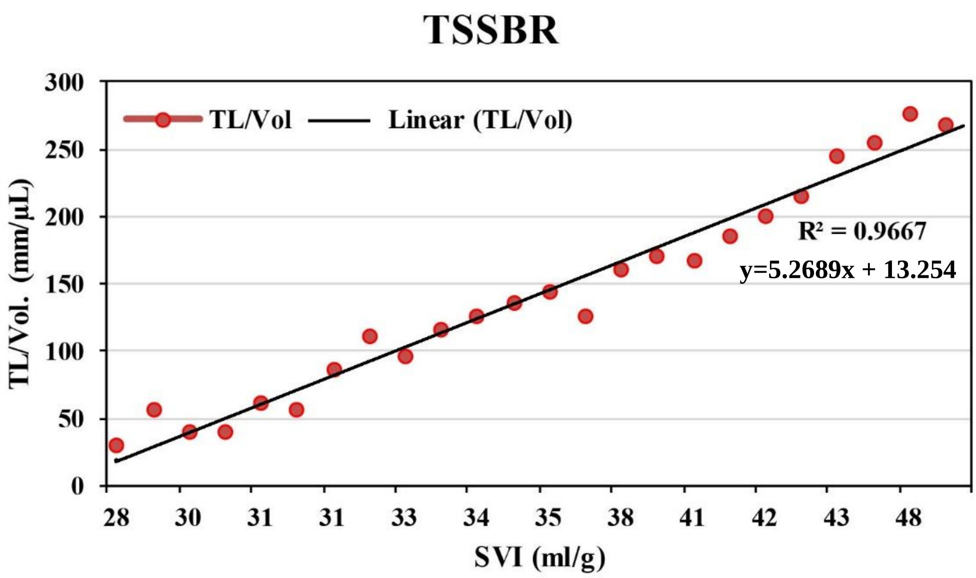



Figure 4.13: filament total length per the sample volume vs. SVI for TSSBR
Source (Alattabi et al., 2017c)

During the first month, there was no clear difference between the morphological characteristics of TSSBR and NOSBR, as seen in Figure 4.14. However, in the second and third months, the settling ability of the NOSBR dropped due to the filamentous growth inside the reactor which can be clearly seen in Figure 4.14., while the morphological characteristics of the sludge inside the TSSBR reactor, have better and more homogenous growth of filamentous bacteria as also seen in Figure 4.14.

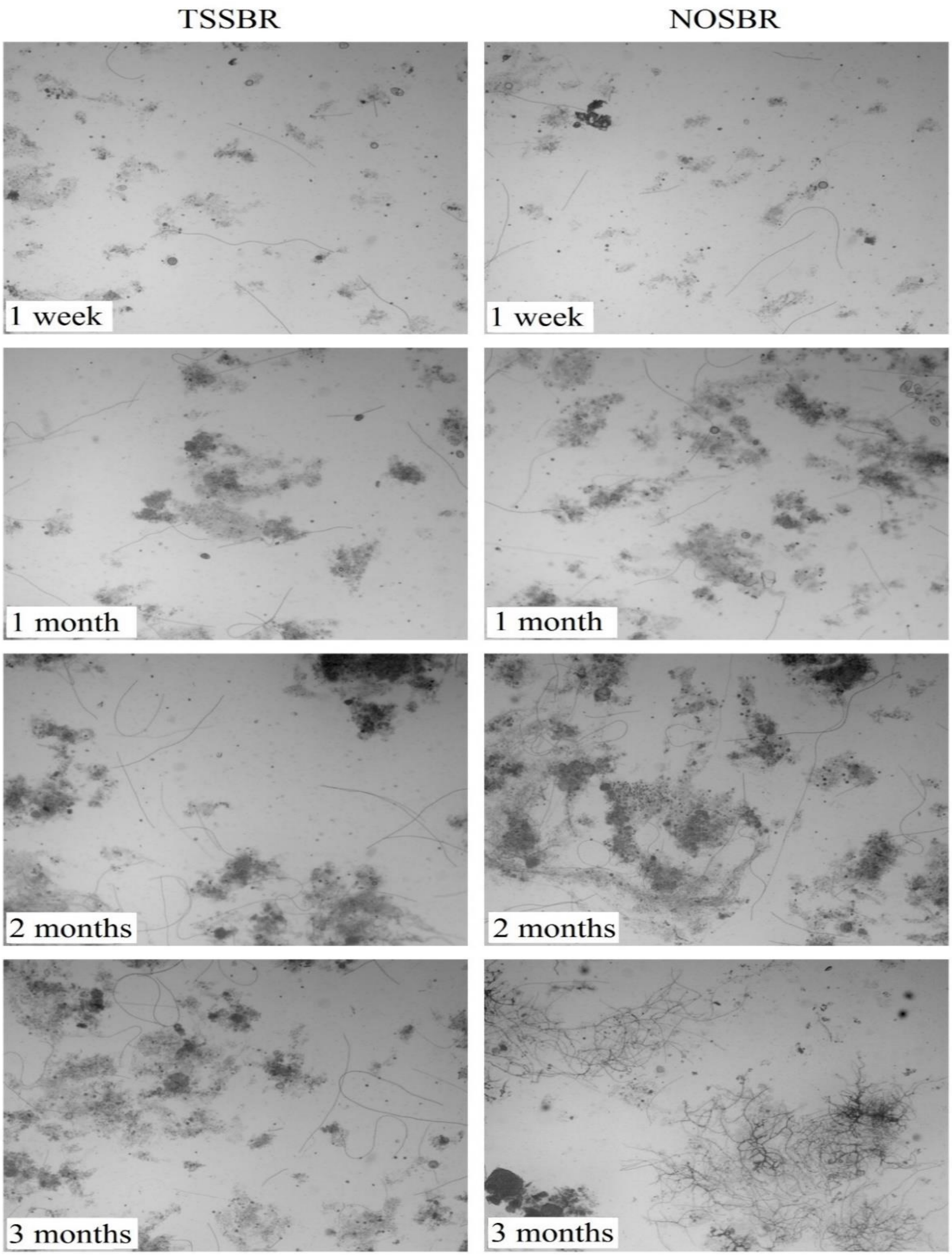



Figure 4.14: 100x microscopic images of sludge sample for NOSBR and TSSBR during different ages (1 week, 1 month, 2 months and 3 months)
Source (Alattabi et al., 2017c)

Over the three-month operation time, the sludge age was kept at between 12-18 days, the MLSS concentration between 2500 mg/l and 3500 mg/l for both reactors (NOSBR and TSSBR). There are two potential reasons for the improvements seen in the TSSBR. The first could be due to breaking down the long settling stage into two stages and producing a shock in the mixing stage after the first settling stage. This led to better compaction of settled and non-settled particles as well as the breaking down of filamentous bacteria. In consequence, a better settle was achieved. The mixing (anoxic) stage, between the two settling stages, has been optimised to get the most advantageous mixing time and speed which was 15 min and 300 rpm, respectively. This was in agreement with Mata et al. (2015) who reduced the settling time by 20% thus giving a decrease in SVI from 325 ml/g to 67 ml/g. In the same vein, Guo et al. (2014b) achieved a significant sludge settleability with anoxic feeding and recommended a mixing stage to improve settling. Mata et al. (2015) found that SVI values decreased by reducing the settling time and allowing intermittent aeration to provide more air, thus supporting the first reason for improving the settleability in the TSSBR.

The second reason for enhancing the settling performance in the TSSBR could be due to minimisation of the anaerobic environment by breaking down the settling time from one hour into two stages: 15 min and 30 min, separated by a 15 min anoxic stage. This creates a negative effect for filamentous bacteria leading to a halt in its growth and an enhanced settling performance in the TSSBR. This result is in agreement with Guo et al. (2014b) who reported that during low DO concentration (0.5 mg/l), sludge settling declined (SVI> 200 ml/g). Liao et al. (2011) found that flocculation ability improved when increasing the DO level from 1-2.5 mg/l to 3.5-5.5 mg/l, which also supports the second issue, decreasing the long settling stage and increasing the DO level by mixing (anoxic stage).

### 4.2.3 Scanning electron microscopy

SEM results were showing that the NOSBR has more abundant filamentous bacteria, while a lower number of filamentous bacteria have been identified in the TSSBR, this can be seen in the SEM pictures in Figure 4.15. This could be due to the same reason mentioned above.

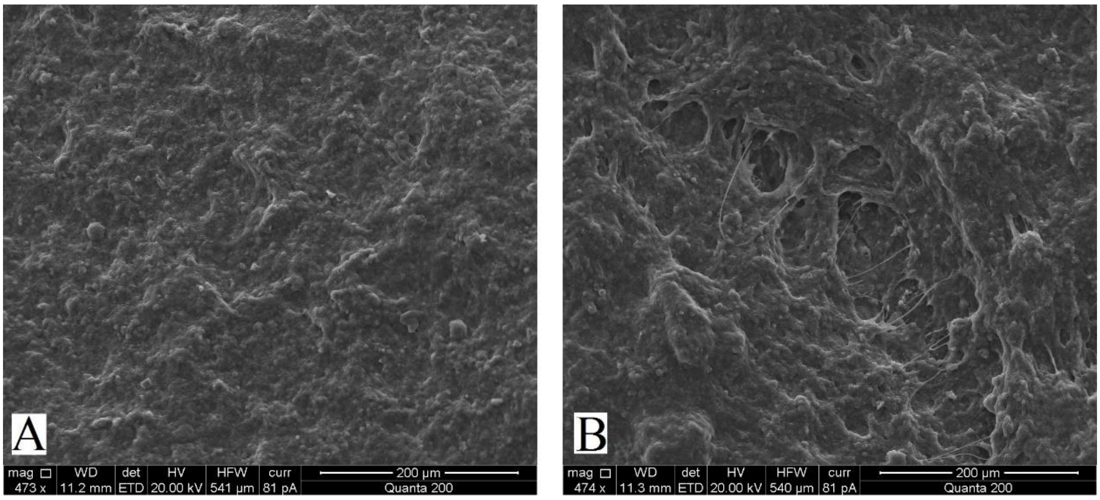



Figure 4.15: SEM images of sludge sample for a) TSSBR and b) NOSBR

## 4.3 Statistical analysis of TSSBR and NOSBR

The results obtained from the statistical analysis confirmed that the performance, regarding SVI and the removal of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N in the TSSBR, is more reliable and predictable than that of NOSBR. It can be seen from the results (Table 4-2) that the standard deviation of the effluent SVI, $NH_3$-N, $NO_3$-N and $NO_2$-N from TSSBR, is much lower than the same effluents in NOSBR, which indicates that TSSBR has better consistency and quality of treatment. In terms of outliers, the results of the statistical analysis (Table 4-2), indicate that the performance of TSSBR is stable as it does not show any extreme readings in effluents SVI, $NH_3$-N, $NO_3$-N and $NO_2$-N. The performance of NOSBR was unstable over the studied period as it showed extreme effluent concentrations of both COD and $NH_3$-N. Finally, in terms of the normality of the obtained results, the Kolmogorov-Smirnov test (ρ of K-S test) indicated that the effluents of both TSSBR and NOSBR followed a normal distribution (ρ of K-S test > 0.05), except for $NO_3$-N from NOSBR, which showed a skewed distribution.

Finally, it should be noted that the calculated mean values of the removal of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N, by TSSBR and NOSBR, confirmed the superior performance of TSSBR (Beta = 0.328, sig = 0.000). It can be seen from Table 4-2 that the SVI value of TSSBR is smaller than that of NOSBR, indicating that TSSBR had better sludge settleability than NOSBR.

Table 4-2: Results of the statistical analysis
Source (Alattabi et al., 2017c)

| Parameter | TSSBR | | | | NOSBR | | | |
|---|---|---|---|---|---|---|---|---|
| | Mean | Standard deviation | Outliers % | ρ of K-S test | Mean | Standard deviation | Outliers % | ρ of K-S test |
| SVI | 39.36 | 10.26 | 0 | 0.210 | 54.86 | 21.14 | 0 | 0.200 |
| COD | 93.51 | 1.061 | 0 | 0.198 | 93.14 | 1.083 | 8.3 | 0.188 |
| $NH_3$-N | 89.24 | 3.42 | 0 | 0.200 | 76.66 | 5.31 | 8.3 | 0.200 |
| $NO_3$-N | 95.24 | 1.38 | 0 | 0.220 | 86.44 | 4.32 | 0 | 0.028 |
| $NO_2$-N | 95.99 | 1.18 | 0 | 0.178 | 87.32 | 2.13 | 0 | 0.169 |

### 4.4 Cost comparison

To obtain the same performances as the TSSBR, the NOSBR was operated for a longer cycle time, and the removal efficiency of nitrogen compounds along with the SVI value were tested every 15 minutes. The removal efficiency of $NH_3$-N of the NOSBR became the same as the TSSBR after increasing the react stage from 240 minutes to 315 minutes as shown in Figure 4.16. Also, the removal efficiency of $NO_3$-N of the NOSBR became the same as the TSSBR after increasing the react stage from 240 minutes to 330 minutes as shown in Figure 4.17. Moreover, the removal efficiency of $NO_2$-N of the NOSBR became the same as the TSSBR after increasing the react stage from 240 minutes to 285 minutes as shown in Figure 4.18. Finally, the SVI values of NOSBR did not reach the SVI values of TSSBR, even when the cycle time of the NOSBR was doubled as shown in Figure 4.19. Thus, the TSSBR system has implied cost benefits compared with the NOSBR despite the additional 15 minutes (anoxic mixing) in the TSSBR cycle of the treatment.

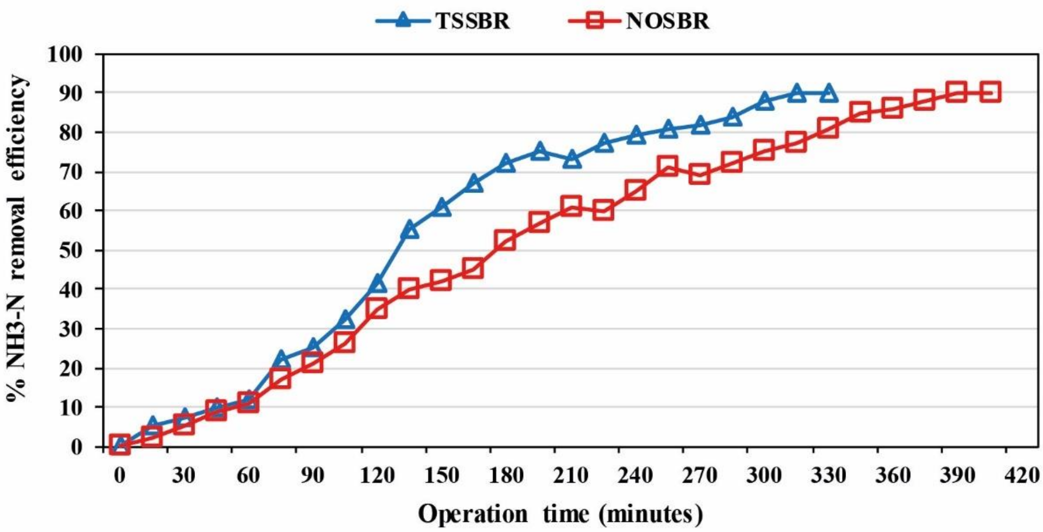



Figure 4.16: $NH_3$-N removal efficiency of NOSBR and TSSBR for cost comparison

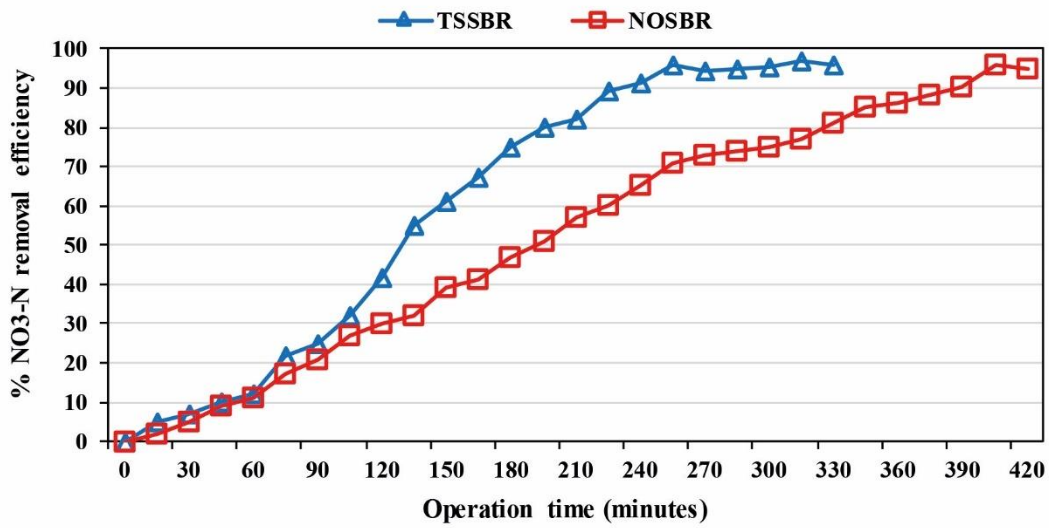



Figure 4.17: $NO_3$-N removal efficiency of NOSBR and TSSBR for cost comparison

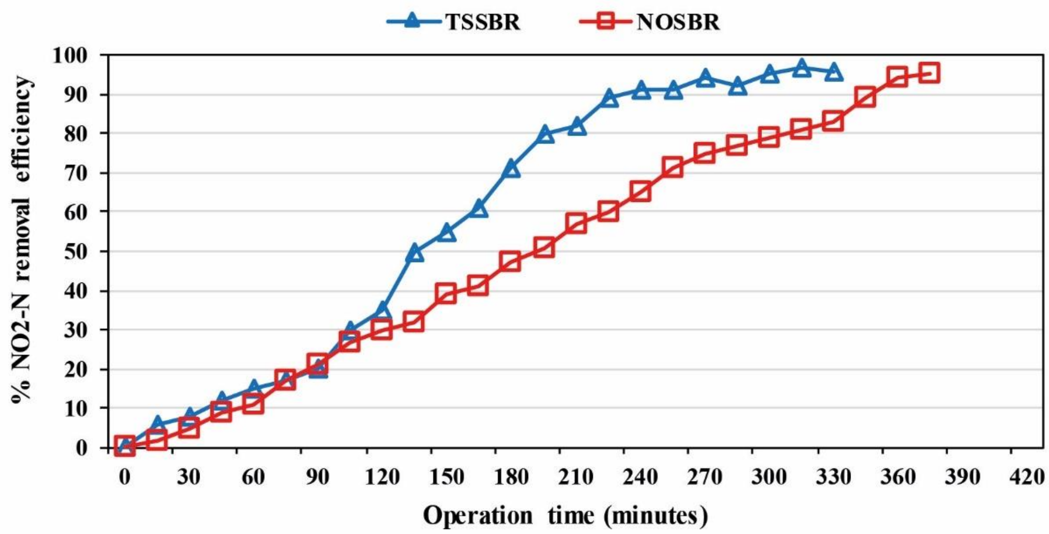



Figure 4.18: $NO_2$-N removal efficiency of NOSBR and TSSBR for cost comparison

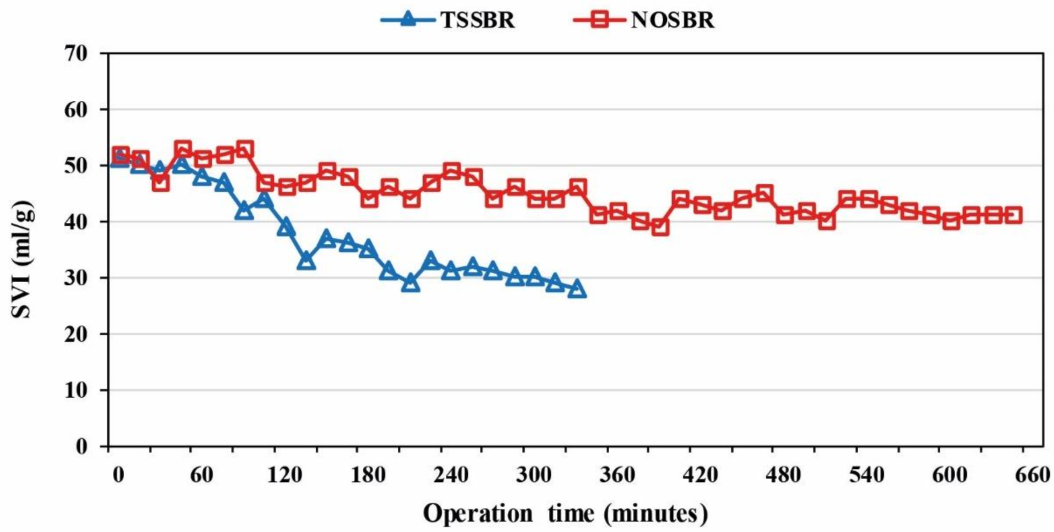



Figure 4.19: Sludge volume index for NOSBR and TSSBR for cost comparison

## 4.5 Chapter summary

In this chapter, the treatment efficiency of both systems (NOSBR and TSSBR) in terms of (COD removal, $NH_3$-N removal, $NO_3$-N removal, $NO_2$-N removal and MLSS) have been discussed. In addition, the sludge settleability of both systems (NOSBR and TSSBR) including (Settled sludge volume, sludge volume index, morphological study, image processing and SEM) have been discussed in this chapter. Moreover, the statistical analysis of the results of both systems (NOSBR and TSSBR) have been discussed in this chapter. Finally, the cost comparison between the NOSBR and TSSBR have been discussed in this chapter.

The summary of the obtained results are listed below, and shown in Table 4-3:

- During the second and third months of operation, the settling ability of the NOSBR dropped due to the filamentous growth inside the reactor, while the morphological characteristics of the sludge inside the TSSBR reactor, have better and more homogenous growth of filamentous bacteria.

- TSSBR system has implied cost benefits compared with the NOSBR despite the additional 15 minutes (anoxic mixing) in the TSSBR cycle of the treatment.

- The results obtained from the statistical analysis confirmed that the performance, in terms of SVI and the removal of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N in the TSSBR, is more reliable and predictable than that of NOSBR.

Table 4-3: TSSBR and NOSBR results summary

| Parameter | Average removal (%) | | Average effluent (mg/l) | |
|---|---|---|---|---|
| | TSSBR | NOSBR | TSSBR | NOSBR |
| COD | 93.1 | 93.7 | 54.7 | 54.83 |
| NH3-N | 89.2 | 76.6 | 0.85 | 1.87 |
| NO3-N | 95.2 | 86.4 | 0.81 | 2.41 |
| NO2-N | 96 | 87.3 | 0.75 | 2.23 |
| | TSSBR | | NOSBR | |
| SVI | 31.17 mg/l | | 42.04 mg/l | |
| TL/MLSS | 1475.33 mm/mg | | 1594.34 mm/mg | |
| TL/Vol | 139.70 mm/µl | | 221.79 mm/µl | |

The TSSBR system proves to be more efficient than NOSBR by improving the sludge settleability and enhancing nitrogen compounds' removal efficiency. Therefore, the TSSBR operating conditions will be optimised in the next chapter (Chapter 5) to get the optimal performance of the system regarding the treatment efficiency and sludge settling performance.

# CHAPTER 5

# TSSBR optimal operation

To achieve the optimal performance of the TSSBR, the operation conditions including (mixed liquor suspended solids, hydraulic retention time, fill conditions, volumetric exchange rate, organic loading rate and hydraulic shock) have been studied, and their effects on the removal efficiency and settling performance are discussed below. Table 5-1 shows the operation values before and during every stage of the optimisation. The importance of the operating parameter was the key for the optimisation order, and the operation values before the optimisation have been chosen based on the literature (Arnz et al., 2000; Sato et al., 2016; Thakur et al., 2013b; Tsang et al., 2007).

Table 5-1: The operation values before and during every stage of the optimisation

| Stage | Description | MLSS (g/l) | HRT (hrs) | Fill modes | Fill time (min) | VER (%) | Glucose OLR (mg/l) | KNO3 OLR (mg/l) |
|---|---|---|---|---|---|---|---|---|
| Stage 0 | Before optimisation | 3 | 5.5 | Aerated fill | 15 | 30 | 1000 | 100 |
| Stage 1 | MLSS optimisation | 2, 3, 4 & 6 | 5.5 | Aerated fill | 15 | 30 | 1000 | 100 |
| Stage 2 | HRT optimisation | 4 | 4, 6, 8 & 12 | Aerated fill | 15 | 30 | 1000 | 100 |
| Stage 3 | Fill modes optimisation | 4 | 5.5 | Aerated & Unaerated | 15 | 30 | 1000 | 100 |
| Stage 4 | Fill time optimisation | 4 | 5.5 | Unaerated fill | 5, 10, 15 & 30 | 30 | 1000 | 100 |
| Stage 5 | VER optimisation | 4 | 5.5 | Unaerated fill | 15 | 20, 40, 60 & 70 | 1000 | 100 |
| Stage 6 | Glucose OLR optimisation | 4 | 5.5 | Unaerated fill | 15 | 20 | 750, 1000, 1250 & 1500 | 100 |
| Stage 7 | KNO3 OLR optimisation | 4 | 5.5 | Unaerated fill | 15 | 20 | 1000 | 50, 100, 150 & 200 |

## 5.1 TSSBR operating conditions optimisation

### 5.1.1 Mixed liquor suspended solids

The four treatment reactors of the TSSBR system were operated under different MLSS concentrations (2, 3, 4 and 6 g/l). The effects of MLSS on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency, settling performance test and microscopic study with image processing using MATLAB and SEM) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.1.1 The effect of MLSS on COD removal efficiency

The effect of concentration of MLSS on COD removal is shown in Figure 5.1. An MLSS concentration of 2 g/l was not very effective because there were not enough microorganisms present to biodegrade all the organic matter in the influent wastewater. Increasing MLSS from 2 to 3 g/l improved the removal efficiency for COD from 89.1% to 92.8%.

Increasing the MLSS further from 3 to 4 g/l did not significantly impact on removal efficiency, measured at 93%. Increasing the MLSS further from 4 to 6 g/l reduced COD removal efficiency to 82.7%. Higher concentrations of MLSS reduce the SBR's performance in relation to organic degradation because the rise in MLSS decreases the food/microorganisms (F/M) ratio, in turn reducing microorganism activity.

These results are in agreement with those of Wanner et al. (Wanner et al., 1998), who found that the removal efficiency of COD was proportionally related to the concentration of MLSS. Watanabe et al. (1994) found that increasing the concentration of MLSS from 4.5 to 5 g/l had no impact on the removal efficiency of COD, this was also the case with Tsang et al.(Tsang et al., 2007), who stated that effluent quality drops under high concentrations of MLSS. The results from this study suggest that an MLSS concentration of between 3 and 4 mg/l is the optimum range where COD removal is at its peak in the TSSBR system.

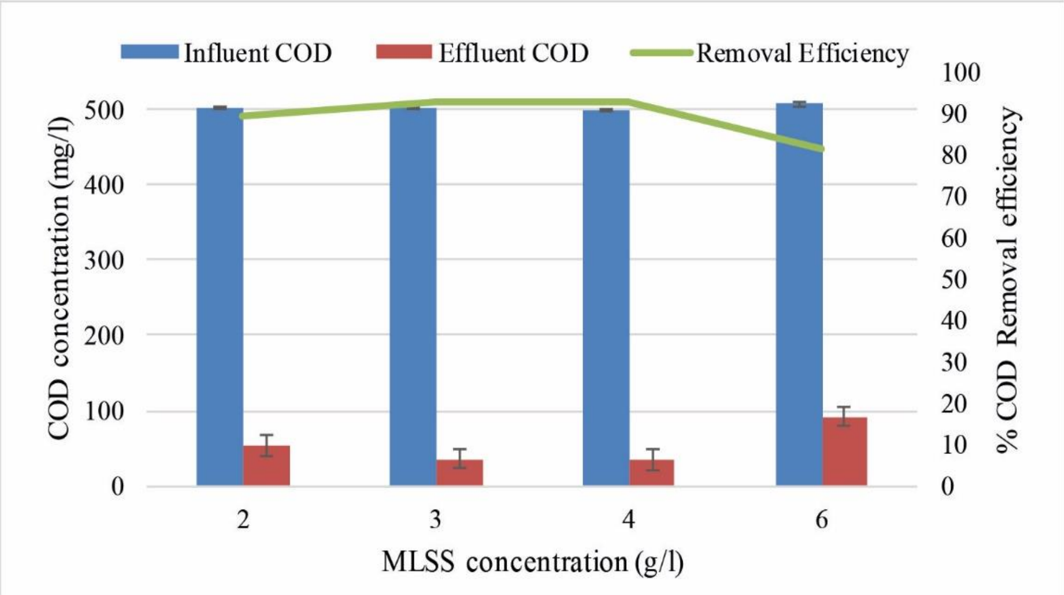



Figure 5.1: The effect of MLSS on COD removal

*HRT 5.5 hrs; Fill 15 min*

### 5.1.1.2 The effect of MLSS on $NH_3$-N removal efficiency

Figure 5.2 shows the removal efficiency of $NH_3$-N along with influent and effluent concentrations with different MLSS concentrations. The removal efficiency of $NH_3$-N was poor at an MLSS concentration of 2 g/l because there were not enough microorganisms present to oxidize all the $NH_3$-N concentrations in the influent wastewater. Increasing the MLSS from 2 to 3 g/l improved the removal efficiency for $NH_3$-N, rising from 87% to 94.6%, with an effluent quality of 0.43 mg/l. Further increasing the MLSS from 3 to 4 g/l did not improve efficiency significantly, measured at 95%. Increasing MLSS from 4 to 6 g/l reduced $NH_3$-N removal efficiency, dropping from 95% to 84%. This reduction in removal efficiency when increasing MLSS is because of an increase in the microorganisms in the system leading to a decrease in the food/microorganisms (F/M) ratio, reducing microorganism activity.

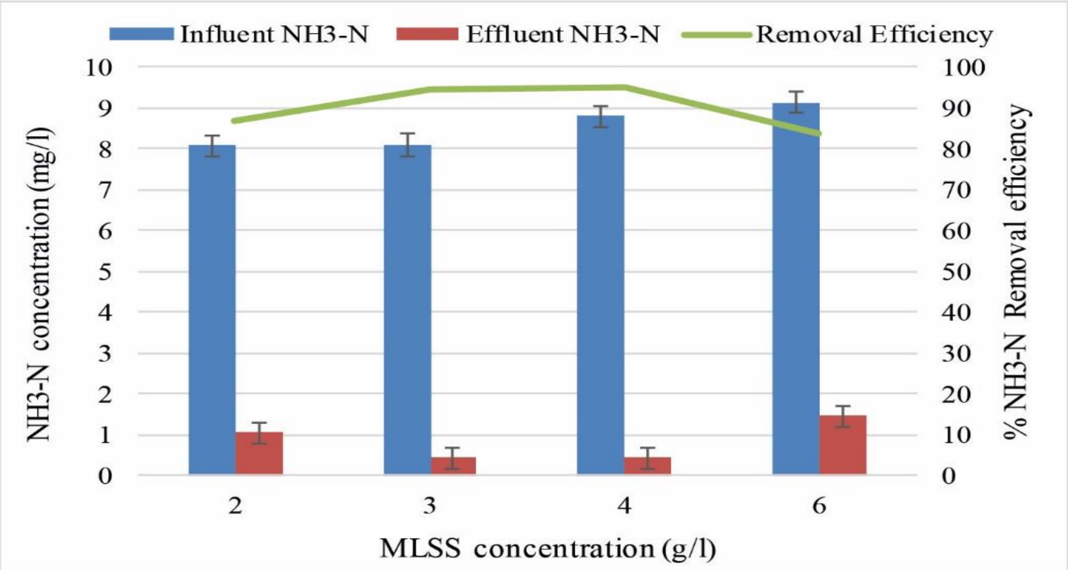



Figure 5.2: The effect of MLSS on $NH_3$-N removal

*HRT 5.5 hrs; Fill 15 min*

#### 5.1.1.3 The effect of MLSS on $NO_3$-N removal efficiency

Figure 5.3 shows the removal efficiency for $NO_3$-N along with influent and effluent concentrations with different concentrations of MLSS. The removal efficiency for $NO_3$-N was poor at an MLSS concentration of 2 g/l suggesting that there were not enough microorganisms present to oxidize all the $NO_3$-N in the influent wastewater. Increasing the MLSS from 2 to 3 g/l improved the removal efficiency from 86.4% to 95.2%, with an effluent quality of 0.85 mg/l. Increasing the MLSS further from 3 to 4 g/l did not affect this, the removal efficiency was measured at 95.6% for 4 g/l of MLSS. Increasing the MLSS further again from 4 to 6 g/l reduced $NO_3$-N removal efficiency, dropping from 95.6% to 80.9% for the same reasons as given above.

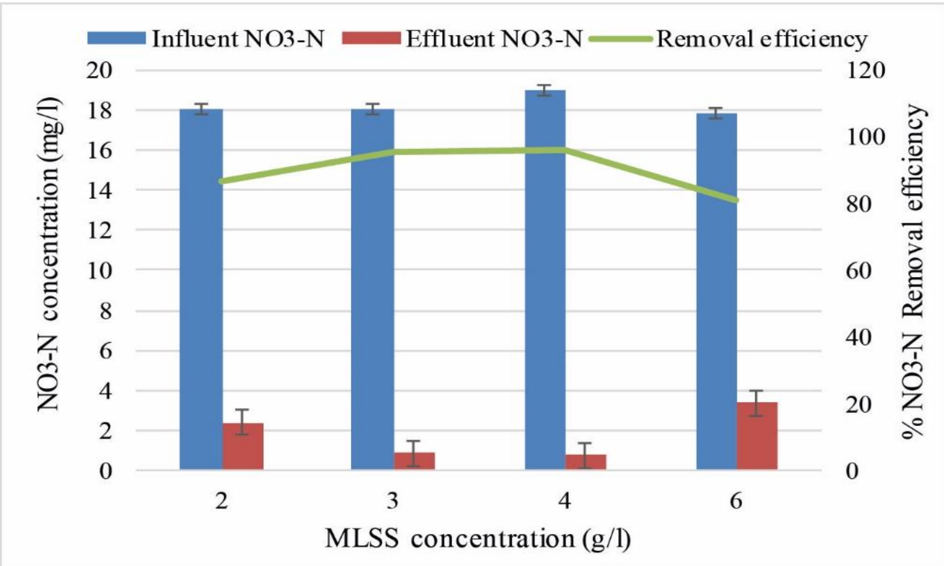



Figure 5.3: The effect of MLSS on $NO_3$-N removal
<u>*HRT 5.5 hrs; Fill 15 min*</u>

Using an aerobic suspension SBR with 24 h HRT, Abu Hasan et al. (Abu Hasan et al., 2016) achieved a 96% removal efficiency for $NH_3$-N and 92.5% removal efficiency for $NO_3$-N. Wei et al. (Wei et al., 2012) achieved a 98% removal efficiency for $NH_3$-

N with an effluent quality of less than 3 mg/l by using a membrane-aerated biofilm reactor (MABR). Chen et al. (Chen et al., 2011) achieved 93.4% removal efficiency for $NH_3$-N with an effluent concentration of 9.4 mg/l, by using an up-flow anaerobic sludge blanket (UASB). The results obtained from this research show that the TSSBR operated with MLSS concentrations between 3 and 4 mg/l with aeration and mixing, offers complete nitrification and denitrification achieving a high removal efficiency of nitrogen compounds.

#### 5.1.1.4 **The effect of MLSS on $NO_2$-N removal efficiency**

Figure 5.4 shows the removal efficiency for $NO_2$-N along with influent and effluent concentrations with different concentrations of MLSS. The removal efficiency for $NO_2$-N was not significant at an MLSS concentration of 2 g/l suggesting that there were not enough microorganisms present to oxidize all the $NO_2$-N in the influent wastewater. Increasing the MLSS from 2 to 3 g/l improved the removal efficiency from 88% to 97.7%, with an effluent quality of 0.42 mg/l. Increasing the MLSS further from 3 to 4 g/l did not affect this, the removal efficiency was measured at 97%. Increasing the MLSS further again from 4 to 6 g/l reduced $NO_2$-N removal efficiency, dropping from 97% to 83% for the same reasons as given above.

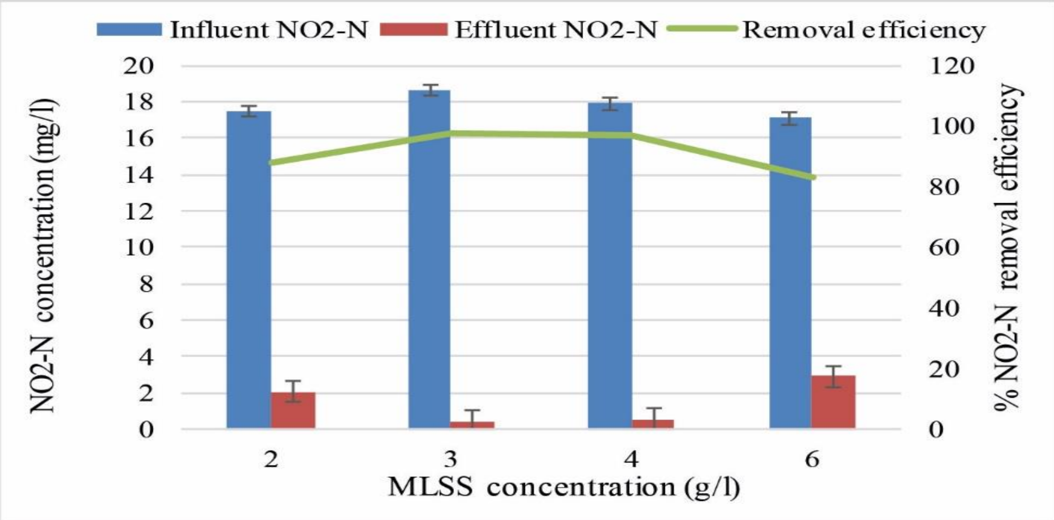



Figure 5.4: The effect of MLSS on $NO_2$-N removal

*HRT 5.5 hrs; Fill 15 min*

The results obtained from this research shows that the TSSBR operated with MLSS concentrations between 3 and 4 mg/l with aeration and mixing, offers complete nitrification and denitrification achieving a high removal efficiency of nitrite-nitrogen.

#### 5.1.1.5 The effect of MLSS on sludge settleability

The impact of MLSS on sludge settling behaviour was studied using four different MLSS concentrations; the SVI was measured regularly to monitor settleability. Figure 5.5 shows the proportional relationship between SVI values and MLSS concentration. At a MLSS concentration of 2 g/l, the best values of SVI were found with an average of 30.1 ml/g, possibly due to the smaller amount of biomass which can settle in the bioreactor system (Tsang et al., 2007). Raising the MLSS from 2 to 3 g/l promoted an increase in SVI values from 30.1 ml/g to 34. 8 ml/g, but thereafter there was no further change even when the concentration of MLSS was increased from 3 to 4 g/l, the SVI measuring 35.2 % at 4 g/l SVI. The SVI value rose to 49.3 ml/g when MLSS was increased from 4 to 6 g/l, possibly because the filamentous bacteria aged when there was an increase in concentration. The results here agree with Tsang et al. (Tsang et al., 2007), who stated that effluent quality was negatively affected by an increase in the concentration of MLSS. They recorded 52.7 ml/g SVI when operating the SBR system with an MLSS concentration of 4.5 g/l, the SVI increasing with increases in MLSS.

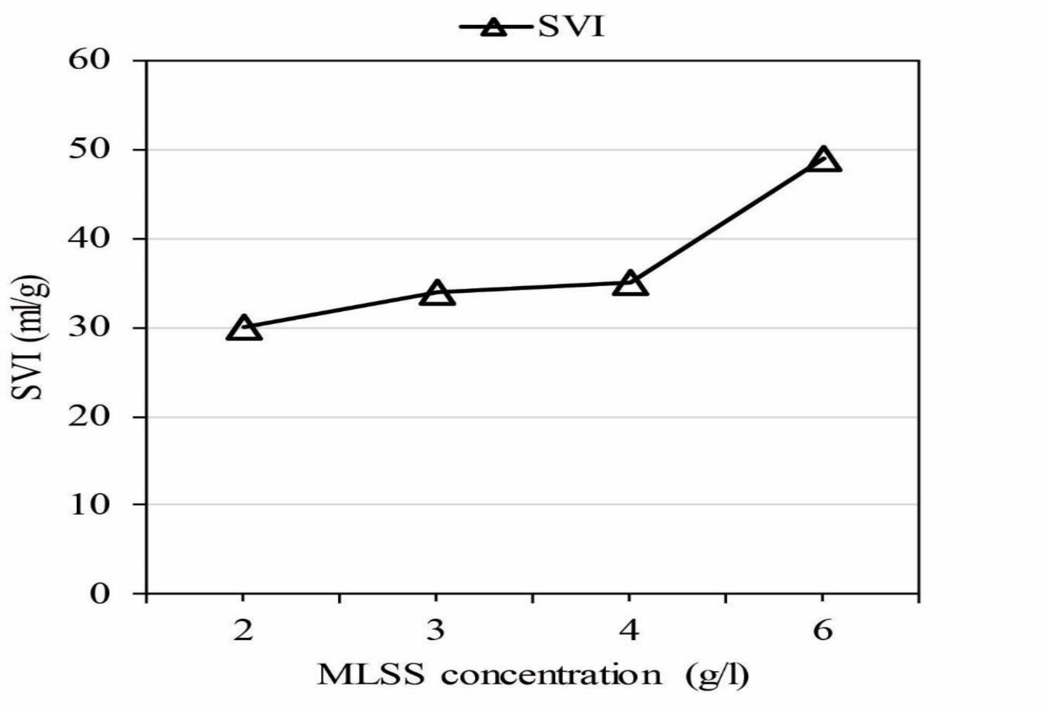



Figure 5.5: The effect of MLSS on SVI value
*HRT 5.5 hrs; Fill 15 min*

Figure 5.6 shows the relationship between filamentous bacteria growth and concentration of MLSS, the results showing that the greater the concentration of MLSS, the more abundant the filamentous bacteria, this can be seen in the SEM pictures in Figure 5.7. This agrees with Da Motta et al. (Da Motta et al., 2002), who studied the effect of filamentous bacteria on settleability through image analysis, finding that filamentous bulking occurred when MLSS was increased in the treatment reactor. Jin et al. (Jin et al., 2003) stated that good sludge settleability could occur when the value of SVI was under 150 ml/g; at that range, filamentous bacteria could appear in low to moderate numbers. Sezgin (1982) also reported a marked increase in SVI when there was an increase in filamentous length in the system. It should be noted however that although the presence of filamentous bacteria in the ASP is desired, an excess amount may cause sludge settling problems (Gerardi, 2002). It can be seen from the results of this study that an MLSS concentration of 2 g/l proved to be the best concentration for solid settling performance, but not the best for effluent quality. A range of MLSS between 3 g/l and 4 g/l is better for TSSBR operation to ensure a good settling performance and to enhance effluent quality at the same time.

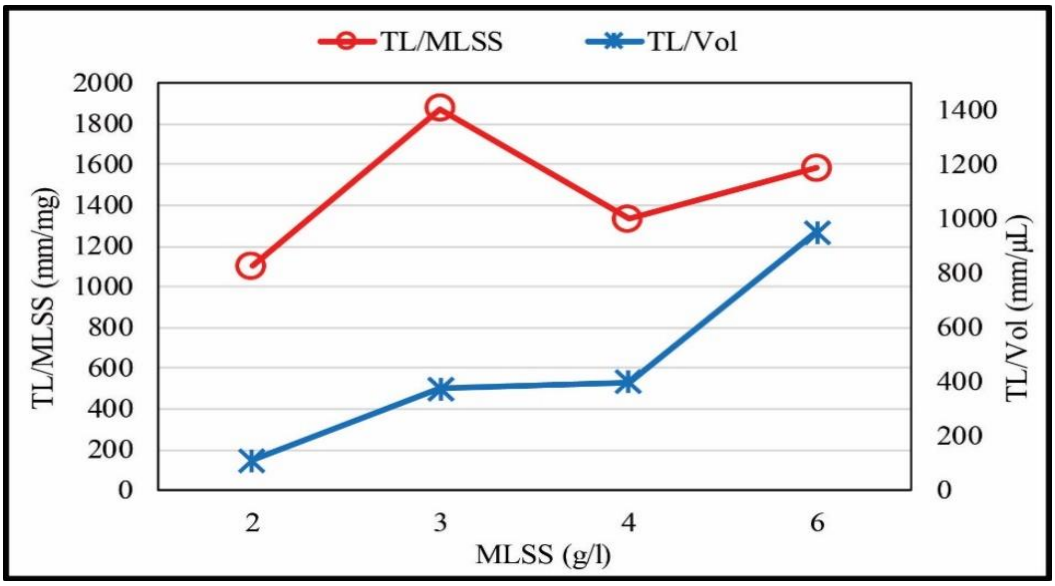



Figure 5.6: The effect of MLSS on Filamentous length (TL/MLSS and TL/Vol)
Source (Alattabi et al., 2017d)

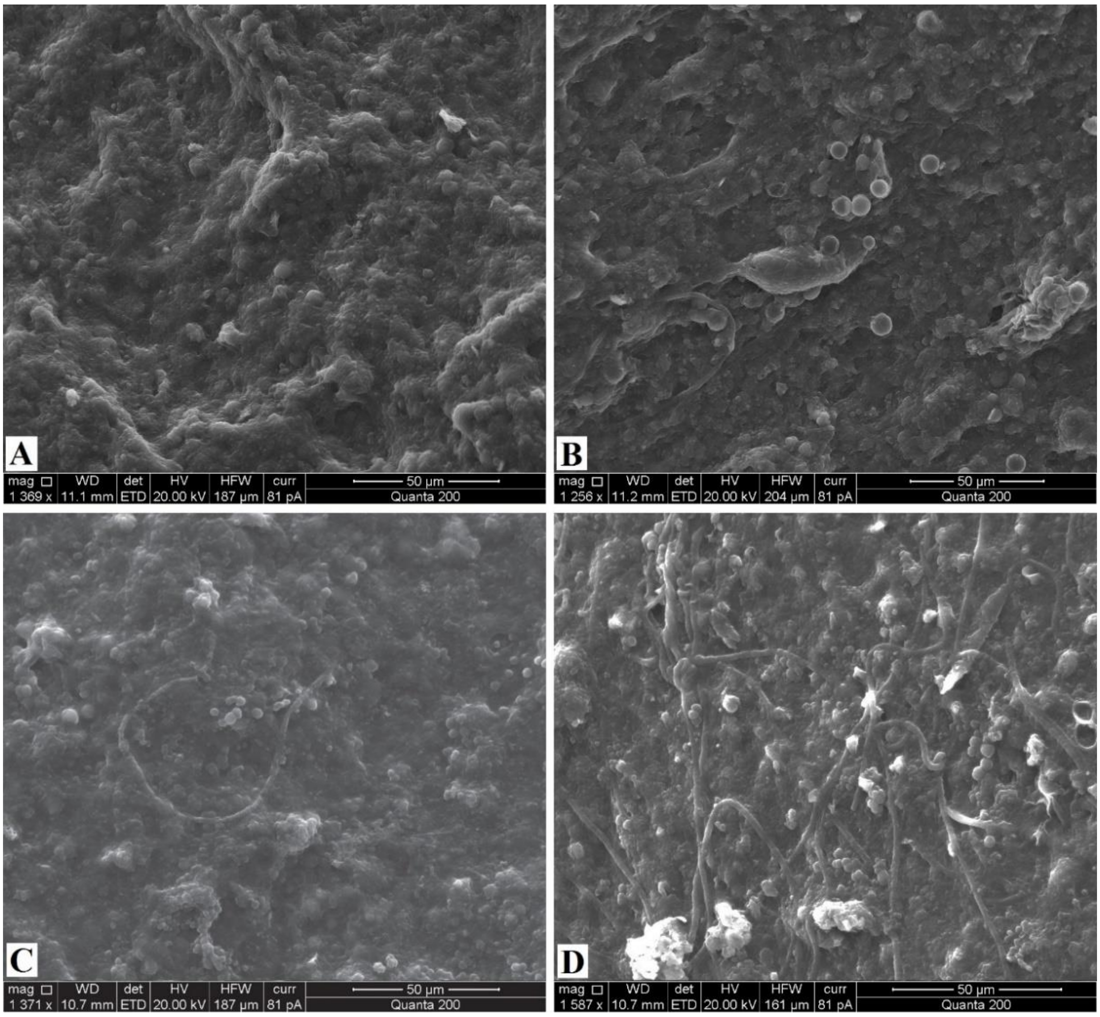



Figure 5.7: SEM image of the sludge (a) 2 g/l MLSS; (b) 3 g/l MLSS; (c) 4 g/l MLSS; (d) 6 g/l MLSS
Source (Alattabi et al., 2017d)

#### 5.1.1.6 pH, ORP, DO and temperature

The monitoring of pH, DO, ORP and temperature under different MLSS concentration is shown in Figures 5.8-5.9. The pH, DO, ORP and temperature values at the end of the 5.5 h treatment cycle fluctuated between 6.5-8.5, 0.4-6 mg/l, -122 to198 mV and 7-15 ◦C, respectively. In the activated sludge process, DO is related to the aerobic stage, while pH and ORP are related to the anoxic and anaerobic stages. The microbial activity in the SBR system is responsible for the variation in the DO profile. Bacteria utilize the DO in the system to oxidize COD and ammonia. At the first stage of the treatment, the anoxic fill, the ORP profile decreases due to the denitrification occurring in the presence of a carbon source in the influent wastewater and anoxic environment (Alattabi et al., 2017a). In the react stage, the aerobic condition, the oxidation of COD begins; this is seen by the increased concentration of ammonia. In this stage, the DO profile increased continuously, while the ORP profile decreased. This might be due to the high concentration of COD in the system. This finding is in agreement with Li and Irvin (2007), who stated that during the anoxic period, ORP dropped to −104 mV under high COD conditions (1317 mg/L), while ORP was still as high as 178mV under low COD conditions (88 mg/L). By 160 minutes into the process, nitrification has started, and this has seen a decrease in ammonia and an increase in nitrite and nitrate concentrations. At this stage, both DO and ORP profiles have increased dramatically (Holman and Wareham, 2005). Denitrification occurred at 225 minutes, identifiably by a decrease in nitrate concentrations. At this stage, DO profile remained constant. At the settle stage, DO concentrations have decreased sharply towards the end of the treatment cycle. There is no clear difference in these profiles under the different MLSS concentration. However, the behaviour of the nutrients removal in each MLSS concentration can be related to these profiles. Based on these results, pH, DO and ORP are considered important parameters that can indicate different behaviours in COD and nitrogen removal.

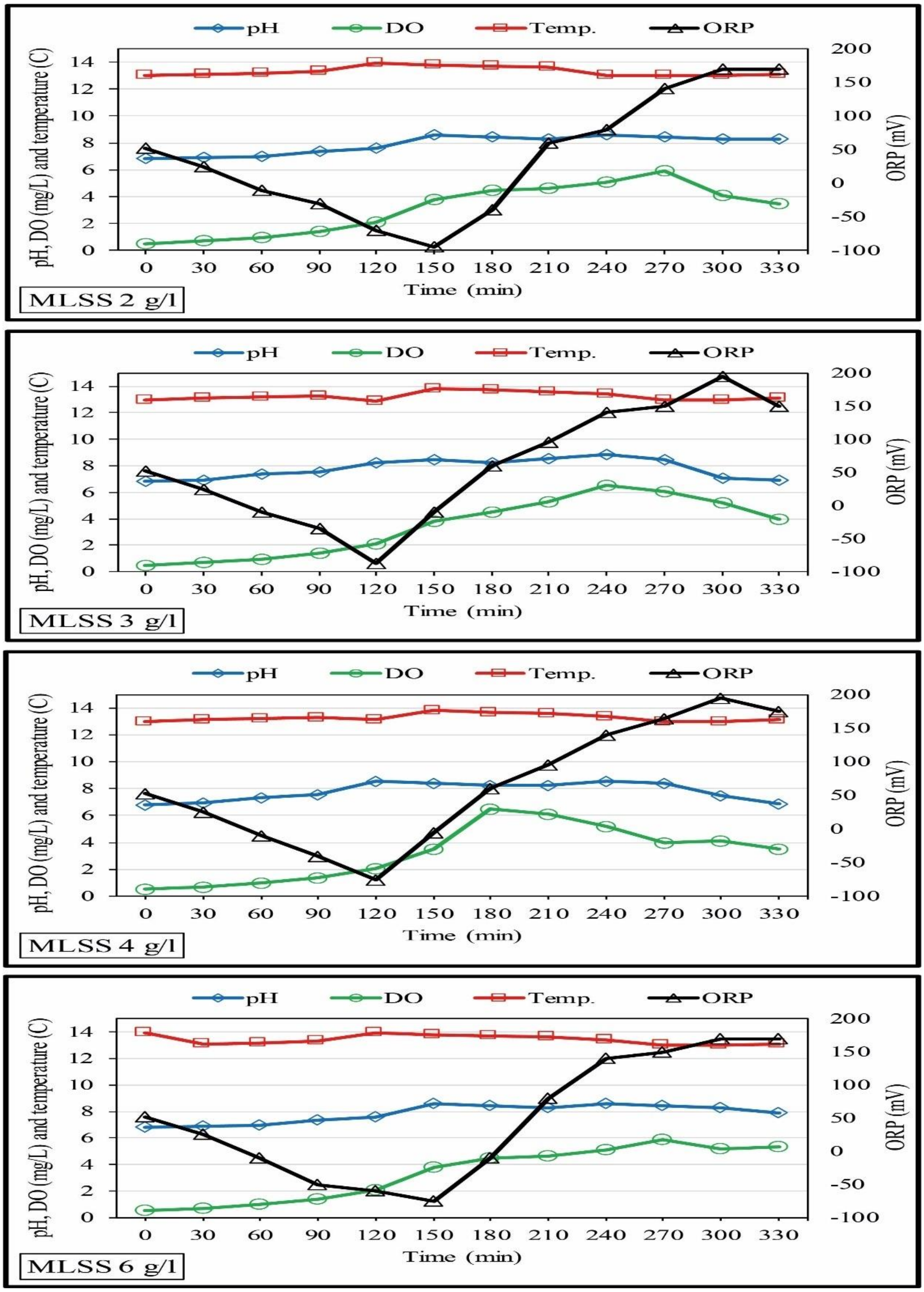



Figure 5.8: pH, DO, temperature and ORP profiles under different MLSS concentration
Source (Alattabi et al., 2017d)

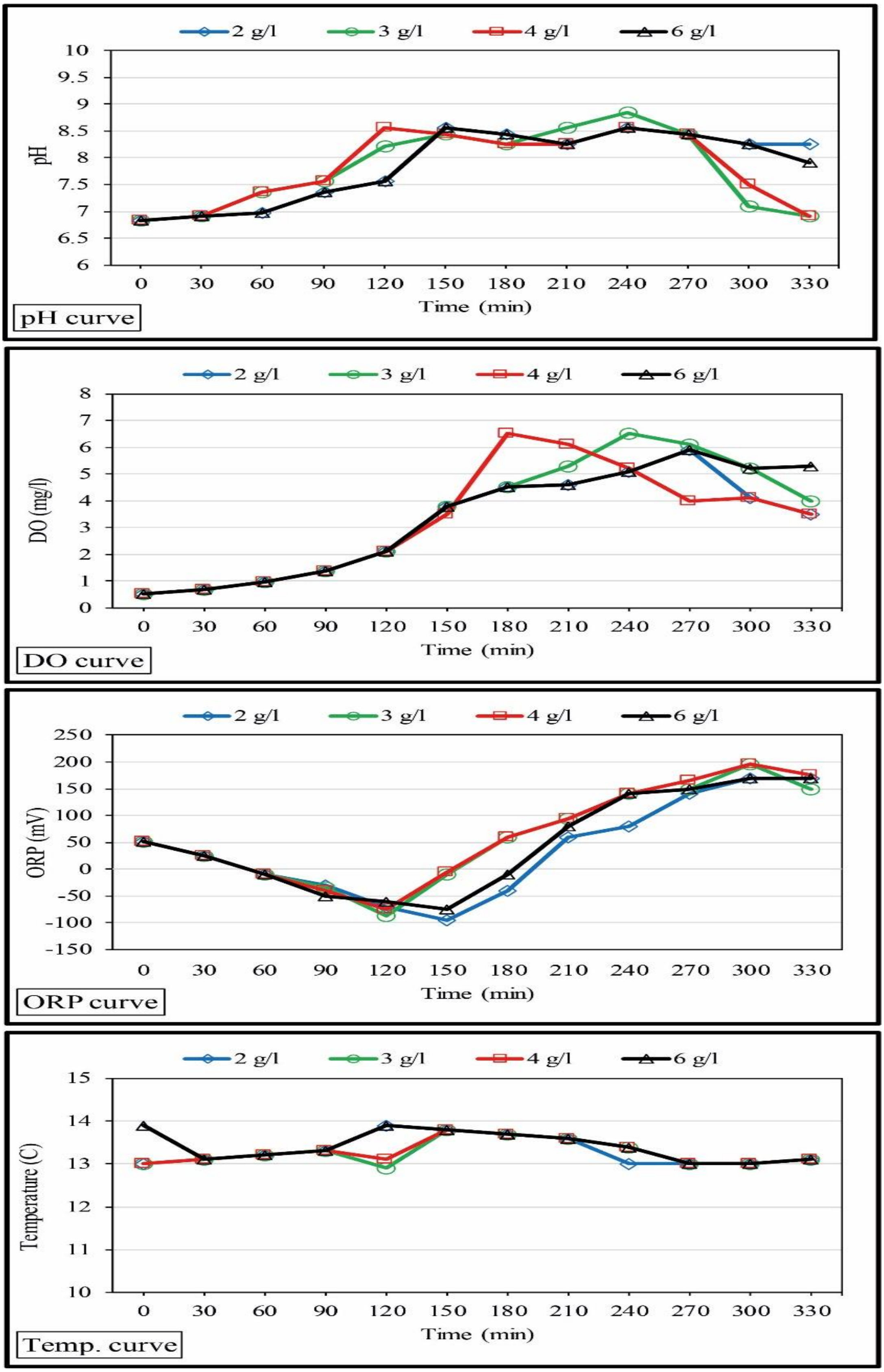



Figure 5.9: pH, DO, temperature and ORP curves under (2, 3, 4 and 6 g/l of MLSS)

### 5.1.2 **Hydraulic retention time**

The four treatment reactors were operated under different HRTs (4, 6, 8 and 12 h). The effects of HRT on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance test) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.2.1 **The Effect of HRT on COD removal**

The effect of HRT on COD removal efficiency is shown in Figure 5.10. It can be seen from the results that the COD removal efficiency has significantly improved when increasing the cycle time from 4 h to 6 h; it was raised from 78% to 94%. This may be because higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). However, the COD removal efficiency decreased when the HRT increased from 6 h to 8 h; it declined from 94% to 91.1%. Moreover, the COD removal efficiency was not affected when increasing the HRT from 8 h to 12 h. This disagrees with (Abu Hasan et al., 2016), who achieved up to 89%, 96% and 92.5% removal efficiency for COD, $NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT, and this might be because of the difference in the laboratory setup of the treatment system.

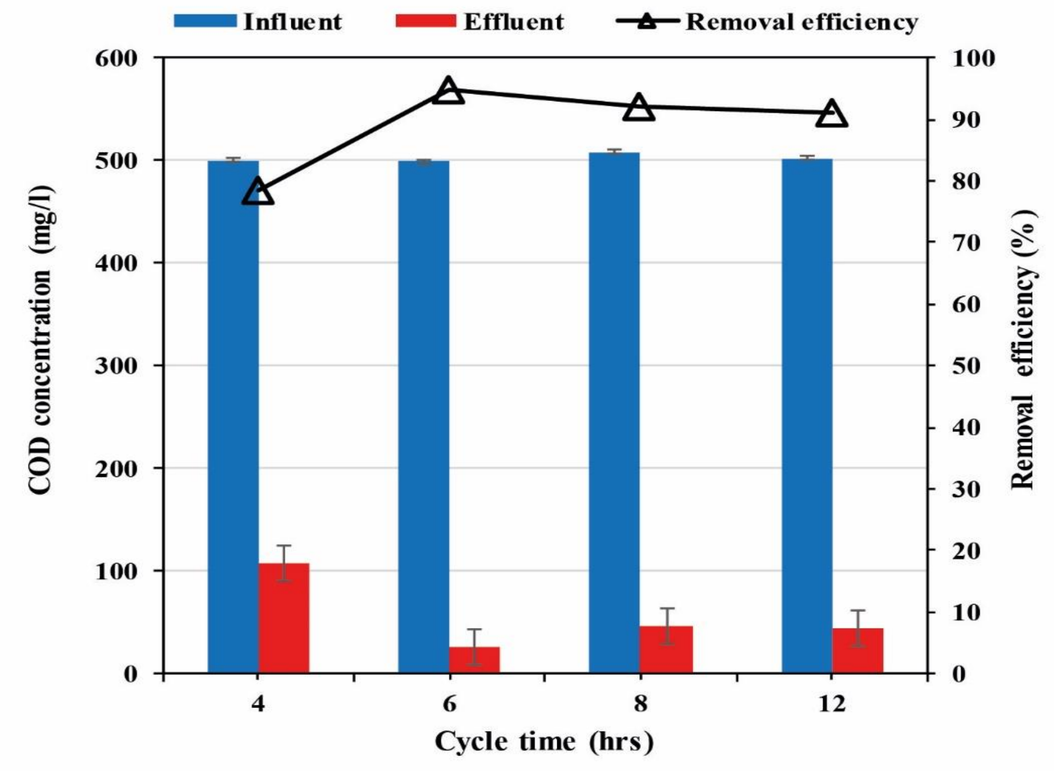



Figure 5.10: The effect of HRT on COD removal

*MLSS 4 g/l; Fill 15 min*

### 5.1.2.2 **The Effect of HRT on $NH_3$-N removal**

The effect of HRT on $NH_3$-N removal efficiency is shown in Figure 5.11. It can be seen from the results that the $NH_3$-N removal efficiency has significantly improved when the cycle time increased from 4 h to 6 h; it was raised from 87% to 94.6%. This may be because higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). However, $NH_3$-N removal efficiency decreased when the HRT increased from 6 h to 8 h; it declined from 94.6% to 86.1%. Moreover, the $NH_3$-N removal efficiency was not affected when the HRT increased from 8 h to 12 h. This disagrees with (Abu Hasan et al., 2016), who achieved up to 89%, 96% and 92.5% removal efficiency for COD,

$NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT, and this might be because of the difference in the laboratory setup of the treatment system.

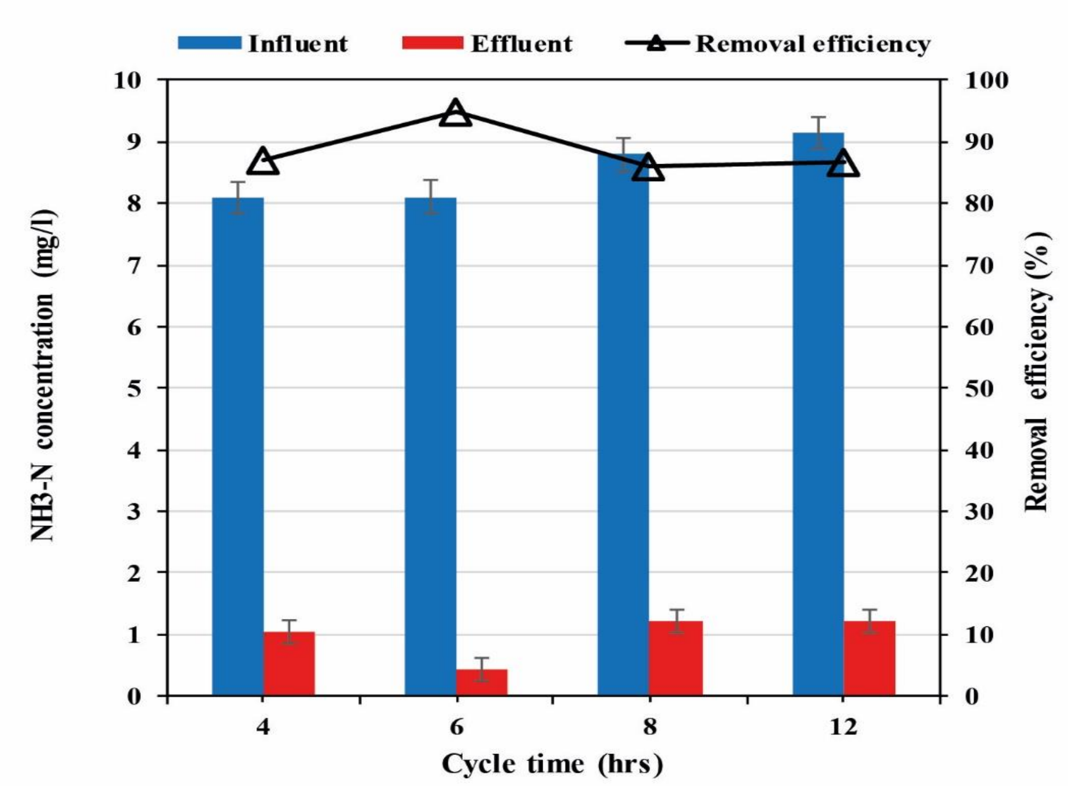



Figure 5.11: The effect of HRT on $NH_3$-N removal

*MLSS 4 g/l; Fill 15 min*

### 5.1.2.3 The Effect of HRT on $NO_3$-N removal

The effect of HRT on $NO_3$-N removal efficiency is shown in Figure 5.12. It can be seen from the results that the $NH_3$-N removal efficiency has significantly improved when the cycle time increased from 4 h to 6 h; it was raised from 76% to 96.8%. This may be because higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). In addition, $NO_3$-N removal efficiency was not affected when the HRT increased from

6 h to 8 h; it was 96.8% for 6 h HRT and 95.3% for 8 h HRT. Moreover, the $NO_3$-N removal efficiency was not affected when the HRT increased from 8 h to 12 h. This disagrees with (Abu Hasan et al., 2016), who achieved up to 89%, 96% and 92.5% removal efficiency for COD, $NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT, and this might be because of the difference in the laboratory setup of the treatment system.

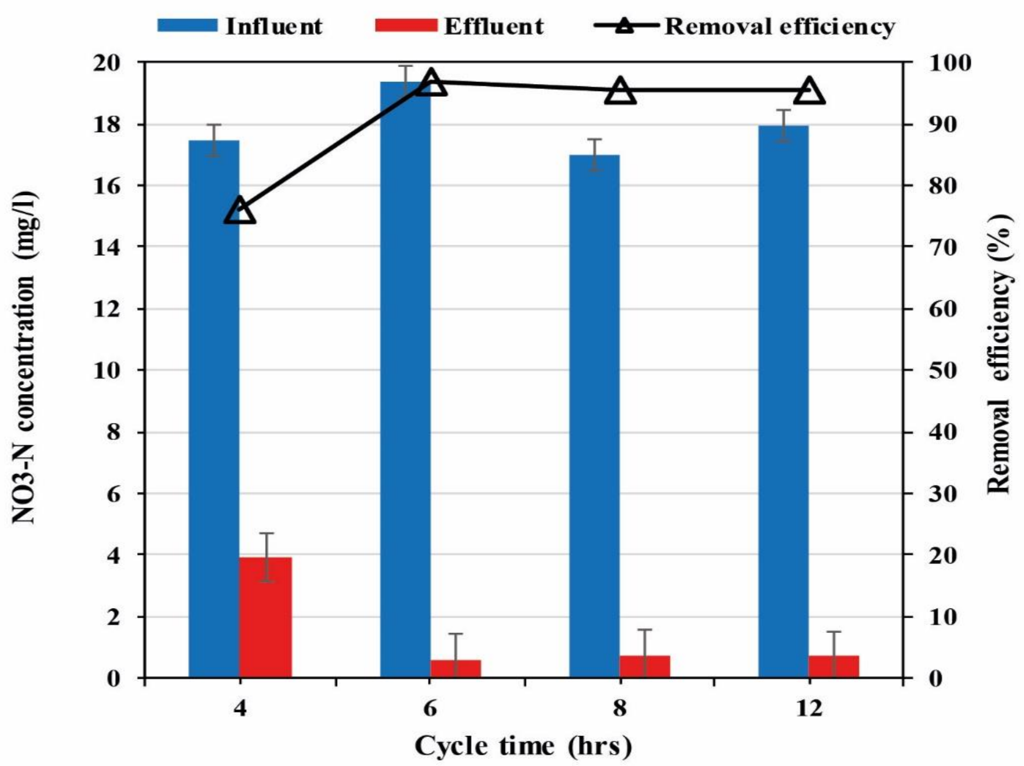



Figure 5.12: The effect of HRT on $NO_3$-N removal
*MLSS 4 g/l; Fill 15 min*

### 5.1.2.4 **The Effect of HRT on $NO_2$-N removal**

The effect of HRT on $NO_2$-N removal efficiency is shown in Figure 5.13. It can be seen from the results that the $NO_2$-N removal efficiency has significantly improved when the cycle time increased from 4 h to 6 h; it was raised from 87% to 96.7%. This may be because higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). However, $NO_2$-N removal efficiency decreased when the HRT increased from 6 h to 8 h; it declined from 96.7% to 91%. Moreover, the $NO_2$-N removal efficiency was not affected when the HRT increased from 8 h to 12 h.

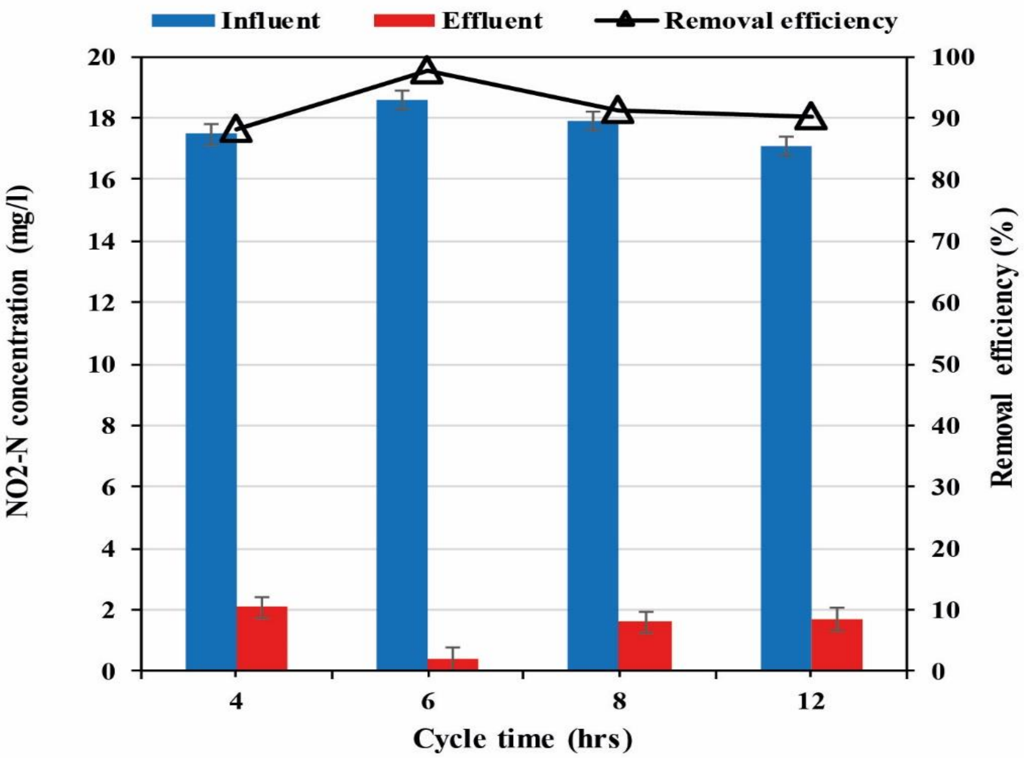



Figure 5.13: The effect of HRT on $NO_2$-N removal

*MLSS 4 g/l; Fill 15 min*

#### 5.1.2.5 **The Effect of HRT on sludge characteristics**

The effect of HRT on sludge characteristics is shown in Figure 5.14. It can be seen from the results that, when the HRT was increased from 4 h to 6 h, this improved the solid settling performance; the SVI value declined from 44.5 ml/g to 35.9 ml/g. Further increasing the HRT to 8 h and 12 h did not affect the settling performance, the SVI values at 8 h and 12 h HRT were 33.3 ml/g and 34.1 ml/g respectively. This agrees with Cervantes (2009), who stated that reducing the HRT will increase the biomass concentration, and thus sludge will take longer to settle.

From the results above, 6 h HRT proves to be the optimal value, in which the removal efficiency of COD, $NH_3$-N, $NO_3$-N, $NO_2$-N and SVI value have been improved dramatically.

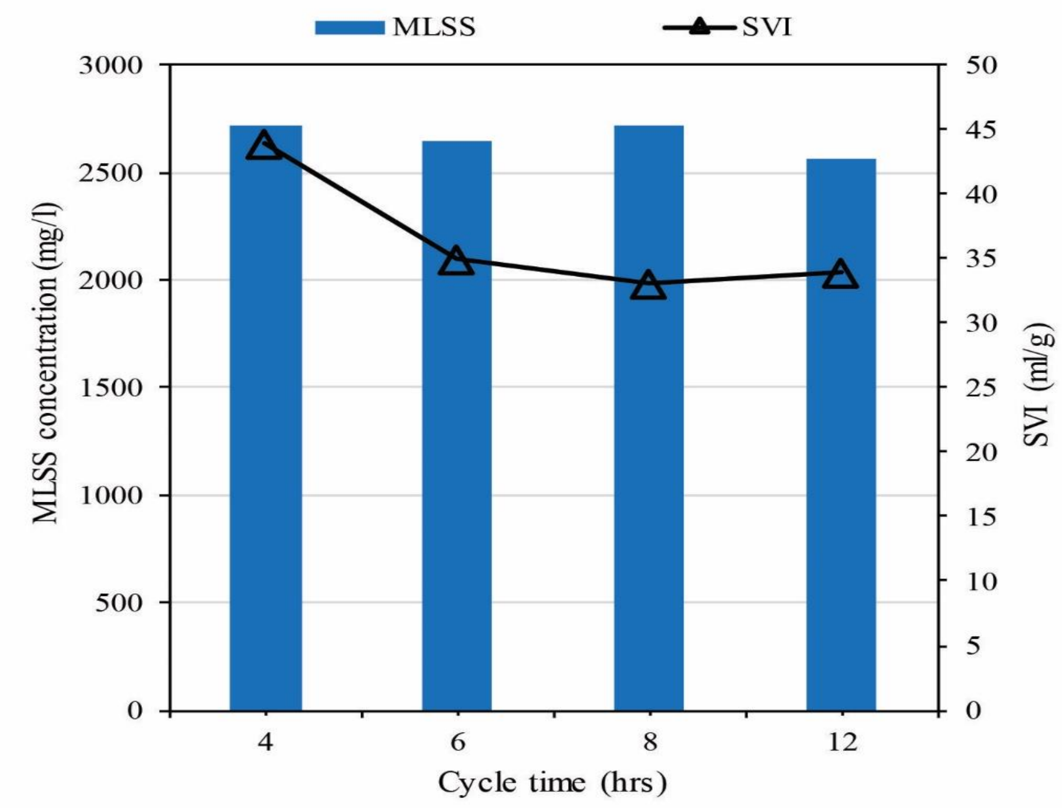



Figure 5.14: The effect of HRT on Sludge characteristics

*MLSS 4 g/l; Fill 15 min*

### 5.1.3 **Fill modes**

Two treatment reactors were operated under two fill modes (aerated fill and un-aerated fill). The effects of fill conditions on both settleability and effluent quality in the TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.3.1 **The Effect of fill modes on COD removal**

The effect of fill modes on COD removal efficiency is shown in Figure 5.15. COD removal efficiency for both aerated and unaerated fill was 91.2% and 90.3% respectively, with an effluent quality of 44 mg COD/l and 48 mg COD/l respectively. The results showed that there is no effect of feeding modes on the treatment efficiency. The two treatment reactors have a high COD removal efficiency.

Chan and Lim (2007) evaluated an SBR performance with aerated and un-aerated fill periods in treating phenol-containing wastewater, the results obtained showed that both periods (aerated and unaerated fill) were capable of maintaining an average phenol removal efficiency of more than 99% even though the influent phenol concentration was increased from 100 to 1000 mg/L. Both (aerated and un-aerated fill) were capable of achieving consistently an effluent quality of less than 1.0 mg/L phenol and 100 mg/L COD concentrations.

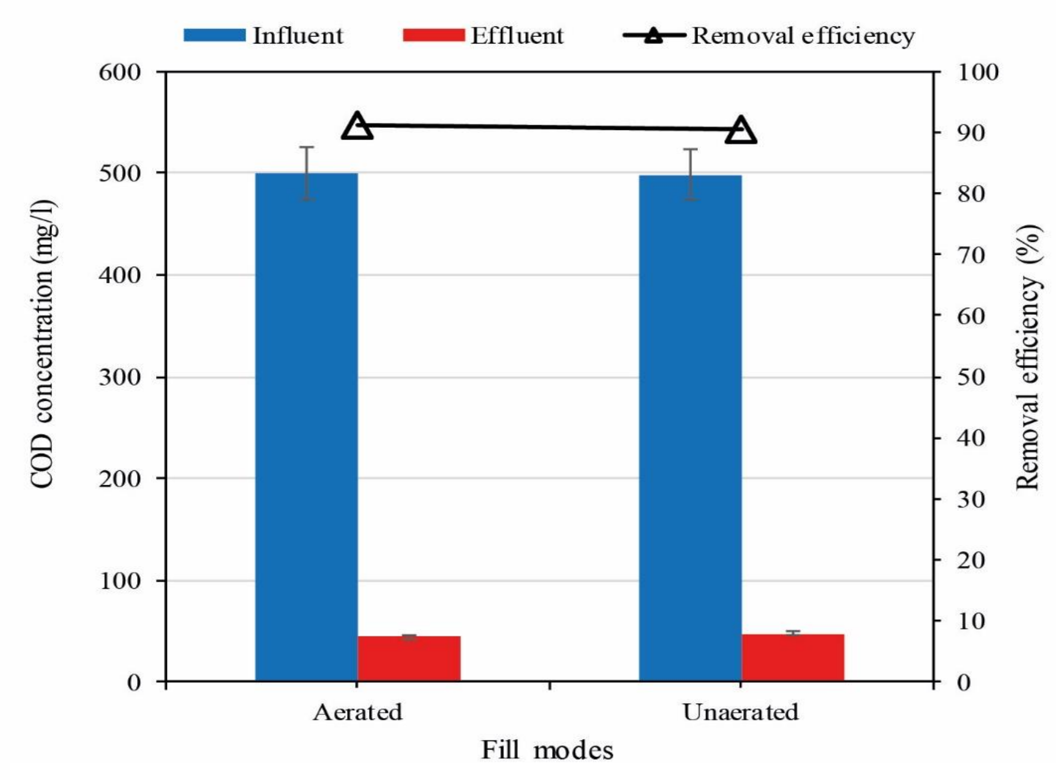



Figure 5.15: The effect of fill modes on COD removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.3.2 **The Effect of fill modes on $NH_3$-N removal**

The effect of fill modes on $NH_3$-N removal efficiency is shown in Figure 5.16. $NH_3$-N removal efficiency for both aerated and un-aerated fill were 88.9% and 89.1% respectively, with an effluent quality of 0.89 mg $NH_3$-N/l and 0.88 mg $NH_3$-N/l respectively. The results showed that there is no effect of feeding modes on the treatment efficiency. This disagreed with Liu et al. (2013) who studied the effect of fill and aeration modes and influent COD/N ratios on the nitrogen removal performance; they stated that un-aerated fill could have relatively higher $NH^{4+}$-N removal due to stronger microbial activity under the anaerobic conditions, and this might be because of the difference in the laboratory setup of the treatment system.

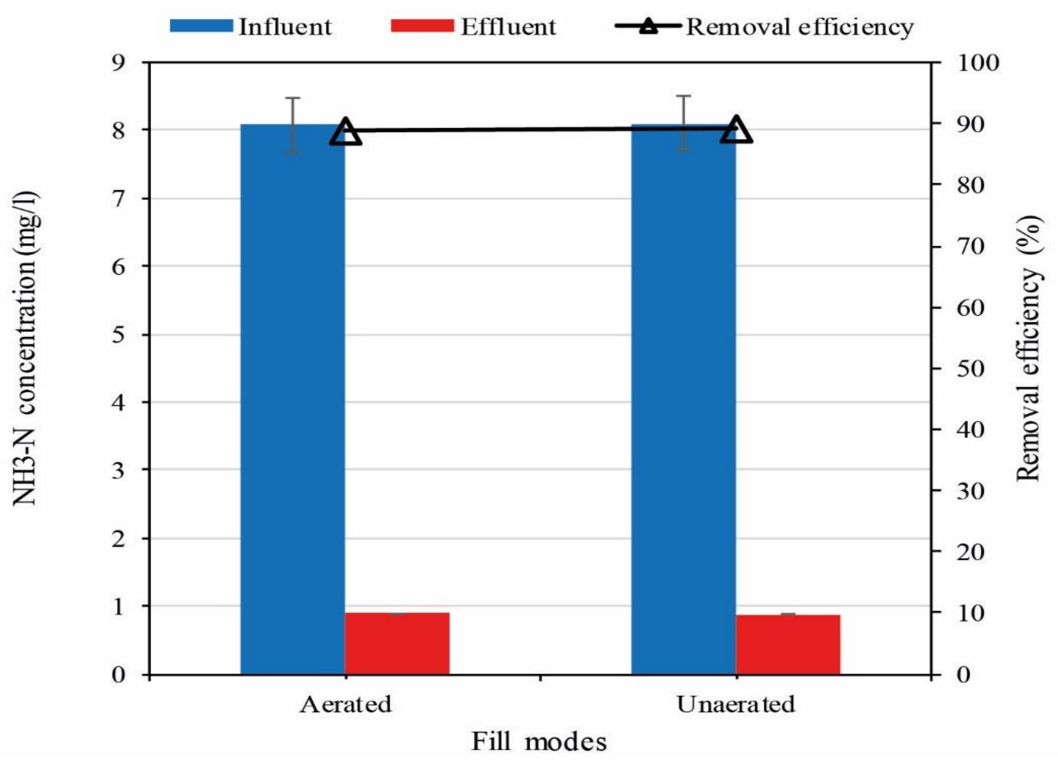



Figure 5.16: The effect of fill modes on $NH_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.3.3 The Effect of fill modes on $NO_3$-N removal

The effect of fill modes on $NO_3$-N removal efficiency is shown in Figure 5.17. $NO_3$-N removal efficiency for both aerated and unaerated fill were 89.4% and 89.1% respectively, with an effluent quality of 1.97 mg $NO_3$-N/l and 2.1 mg $NO_3$-N/l respectively. The results showed that there is no effect of feeding modes on the treatment efficiency. This disagreed with Liu et al. (2013) who studied the effect of fill and aeration modes and influent COD/N ratios on the nitrogen removal performance, and stated that aerated fill could strengthen the nitrogen removal with the presence of carbon source, but no statistically significant effect of intermittent aerated fill on nitrogen removal was observed with the COD/N ratio of 2.5, and this might be because of the difference in the laboratory setup of the treatment system.

In addition, Yu et al. (1996) who studied the effect of fill mode on the performance of sequencing-batch reactors treating various wastewaters, stated that in the aerated fill, the $NO_3$-N concentration was 68 mg/l, while in the un-aerated fill, the $NO_3$-N concentration was only 45 mg/l, based on this, it is clear that there had been an effective denitrification during the un-aerated fill mode probably due to the different availability of organic carbon sources to denitrifiers during the second anoxic mixing period.

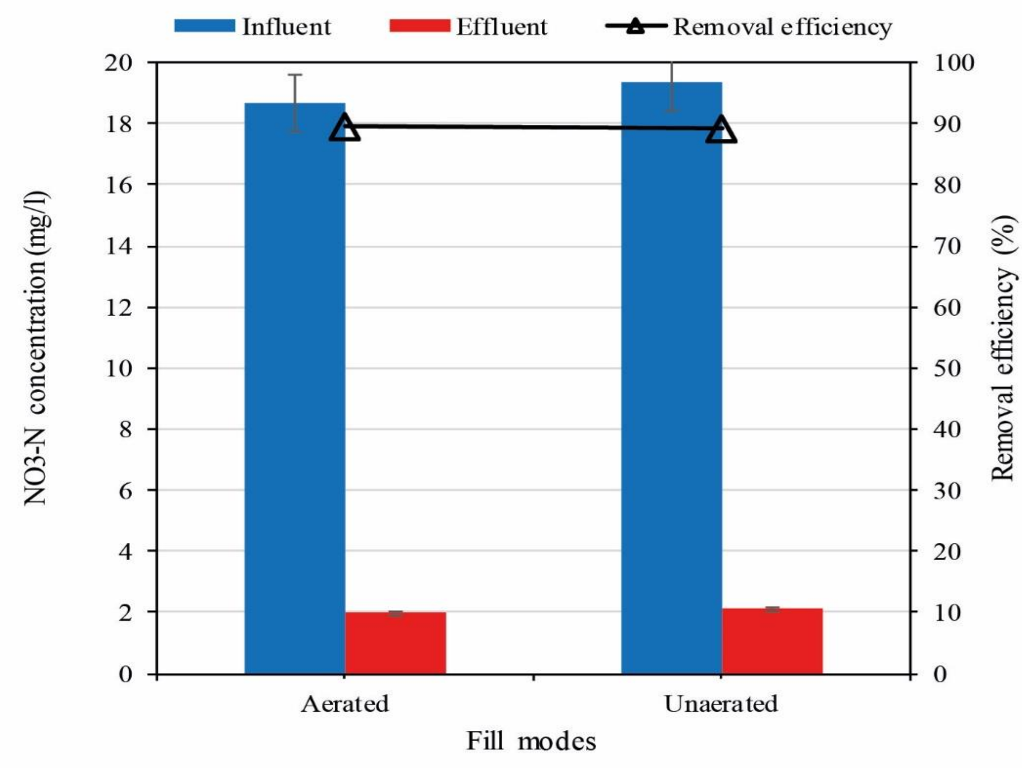



Figure 5.17: The effect of fill modes on $NO_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.3.4 **The Effect of fill modes on $NO_2$-N removal**

The effect of fill modes on $NO_2$-N removal efficiency is shown in Figure 5.18. $NO_2$-N removal efficiency for both aerated and un-aerated fill were 90.7% and 91.1%

respectively, with an effluent quality of 1.62 mg $NO_2$-N/l and 1.65 mg $NO_2$-N/l respectively. The results showed that there is no effect of feeding modes on the treatment efficiency. Again, the results obtained disagreed with Liu et al. (2013) who stated that aerated fill could strengthen the nitrogen removal with the presence of carbon source, but no statistically significant effect of intermittent aerated fill on nitrogen removal was observed. In addition, Yu et al. (1996) stated that both aerated and un-aerated fill were significant in terms of nitrogen compounds' removal rates.

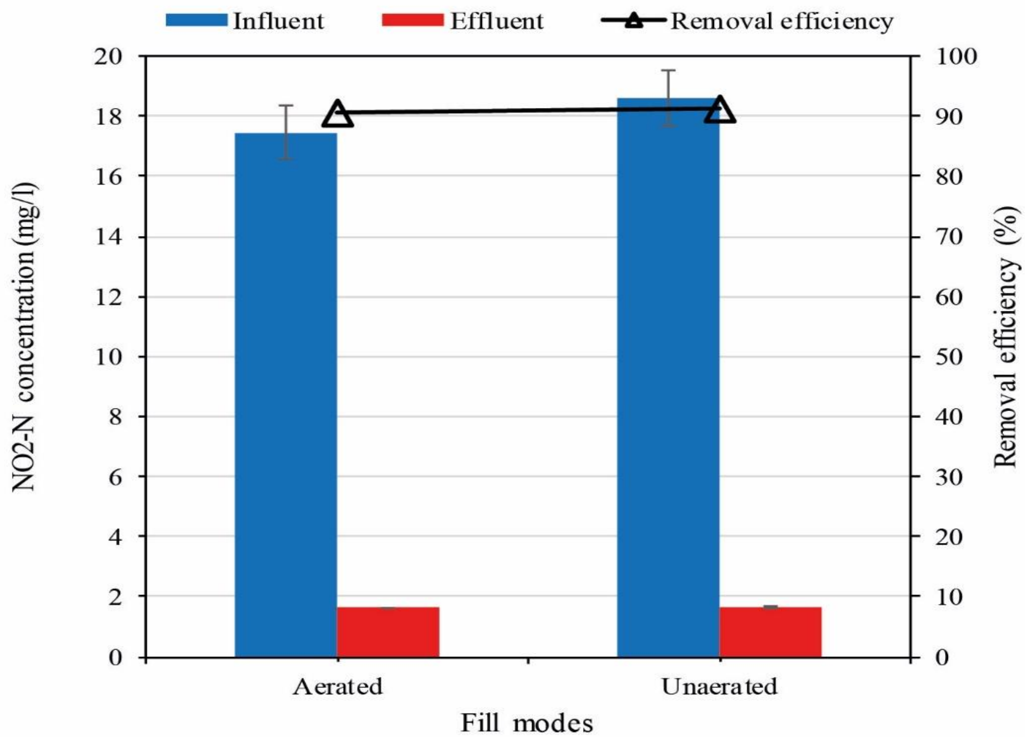



Figure 5.18: The effect of fill modes on $NO_2$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.3.5 **The Effect of fill modes on sludge settleability**

The effect of fill modes on SVI is shown in Figure 5.19. The SVI value was 43.2 ml/g during aerated fill, while it was 34.6 ml/g during un-aerated fill. The improvement in the settling performance in the un-aerated fill over the aerated fill could be because the aeration enhances the growth rate of several kinds of bacteria which would increase the biomass in the reactor and slow down the solids settling performance (Rodriguez-Perez and Fermoso, 2016). Chan and Lim (2007) evaluated an SBR performance with aerated and un-aerated fill periods in treating phenol-containing wastewater, the results obtained showed that the mean SVI values were 93 and 89 mL/g for reactors aerated and un-aerated fill respectively, indicating good sludge settleability when the influent phenol concentration was at 100 mg/L, also, a mean SVI value of 23 mL/g was registered when the influent phenol concentration was at 1000 mg/L, the good sludge settleability in the unaerated fill reactor could be explained by the anaerobic conditions prevailing in the reactor which favoured floc forming organisms.

In addition, while studying the effect of fill mode on the performance of sequencing-batch reactors treating various wastewaters, Yu et al. (1996) stated that at low influent phenol concentrations, the SBR with an un-aerated fill was better than the SBR with an aerated fill in terms of sludge settleability, as aeration encouraged the growth of filamentous bacteria under low substrate concentration conditions. In contrast, when the influent concentration was high, the performance of the latter was superior to the former in which dispersed growth of biomass occurred because of the inhibitory effects of high-strength phenol on microorganisms.

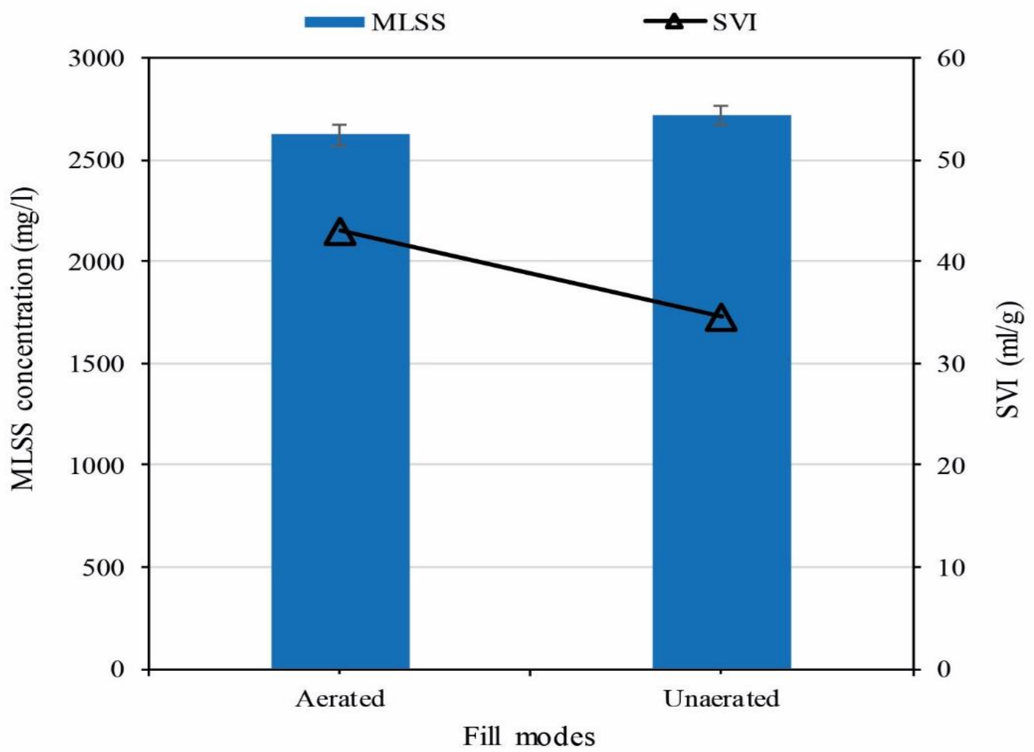



Figure 5.19: The effect of fill modes on sludge characteristics

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.4 **Fill time**

The four treatment reactors were operated under various fill periods (5, 10, 15 and 30 minutes). The effects of fill time on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.4.1 **The Effect of fill time on COD removal**

The effect of fill time on COD removal efficiency is shown in Figure 5.20. The removal efficiency of COD at 5 minutes fill time was 87.7%, when the fill time increased to 10 minutes, there was no significant improve in the removal efficiency of

COD, it was 88.7% at 10 minutes fill time. However, it can be seen from the results that the COD removal efficiency has significantly improved when the fill time increased from 10 minutes to 15 minutes; it was raised from 88.7% to 90.8%. This may be because longer fill time gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). Moreover, the COD removal efficiency was not affected when increasing the fill time increased from 15 minutes to 30 minutes; it was 90.9% at 30 minutes fill time.

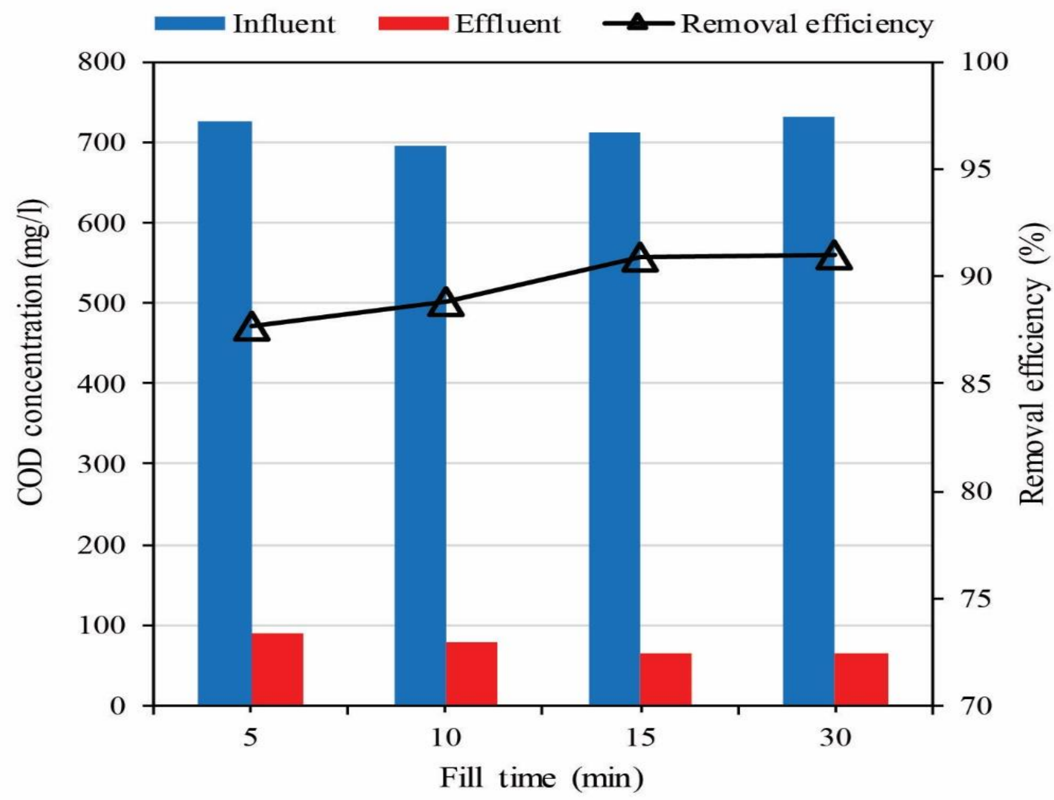



Figure 5.20: The effect of fill time on COD removal

*MLSS 4 g/l; HRT 5.5 hrs*

### 5.1.4.2 **The Effect of fill time on $NH_3$-N removal**

The effect of fill time on $NH_3$-N removal efficiency is shown in Figure 5.21. The removal efficiency of $NH_3$-N at 5 minutes fill time was 84.7%. It can be seen from the results that the $NH_3$-N removal efficiency has significantly improved when the fill time increased from 5 minutes to 10 minutes; it was raised from 84.7% to 87.2%. In addition, $NH_3$-N removal efficiency has significantly improved when the fill time increased from 10 minutes to 15 minutes; it was raised from 87.2% to 90.4%. This may be because longer fill time gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). Moreover, the $NH_3$-N removal efficiency was not affected when the fill time increased from 15 minutes to 30 minutes; it was 90.7% at 30 minutes fill time.

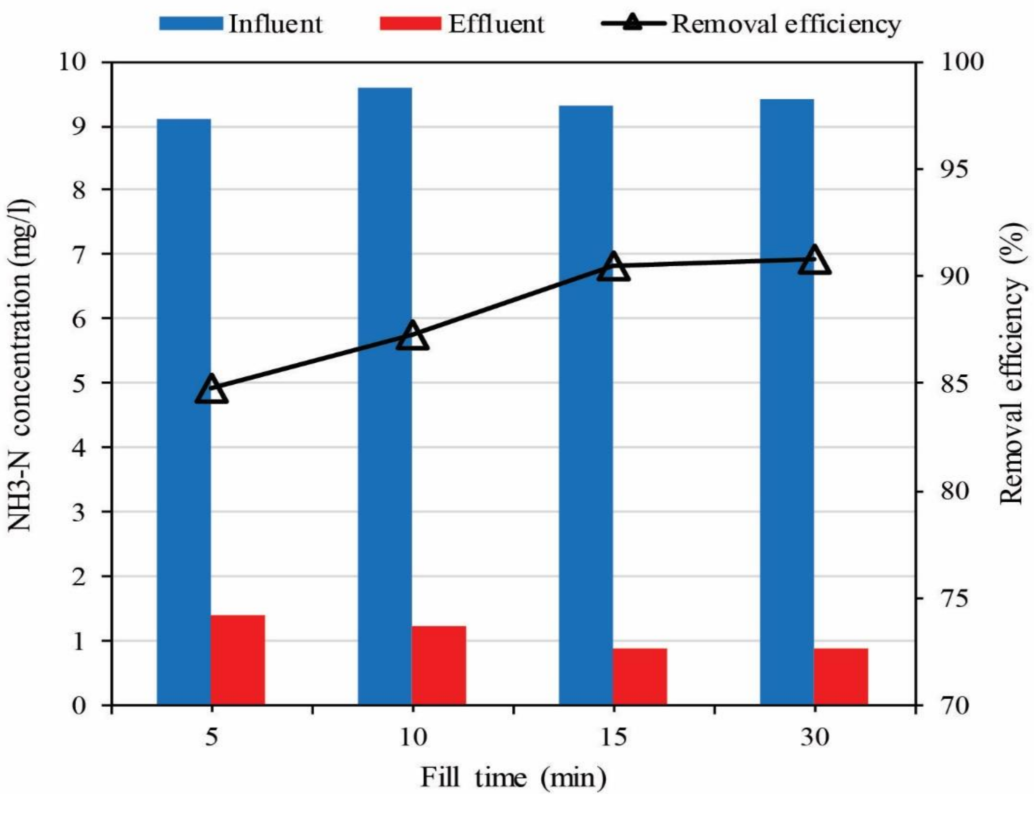



Figure 5.21: The effect of fill time on $NH_3$-N removal

<u>*MLSS 4 g/l; HRT 5.5 hrs*</u>

### 5.1.4.3 **The Effect of fill time on $NO_3$-N removal**

The effect of fill time on $NO_3$-N removal efficiency is shown in Figure 5.22. The removal efficiency of $NO_3$-N at 5 minutes fill time was 90.1%. It can be seen from the results that the $NO_3$-N removal efficiency has significantly improved when the fill time increased from 5 minutes to 10 minutes; it was raised from 90.1% to 92.1%. In addition, $NO_3$-N removal efficiency has improved when the fill time increased from 10 minutes to 15 minutes; it was raised from 92.1% to 94.4%. This may be because longer fill time gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). Moreover, the $NO_3$-N removal efficiency was not affected when the fill time increased from 15 minutes to 30 minutes; it was 94.9% at 30 minutes fill time.

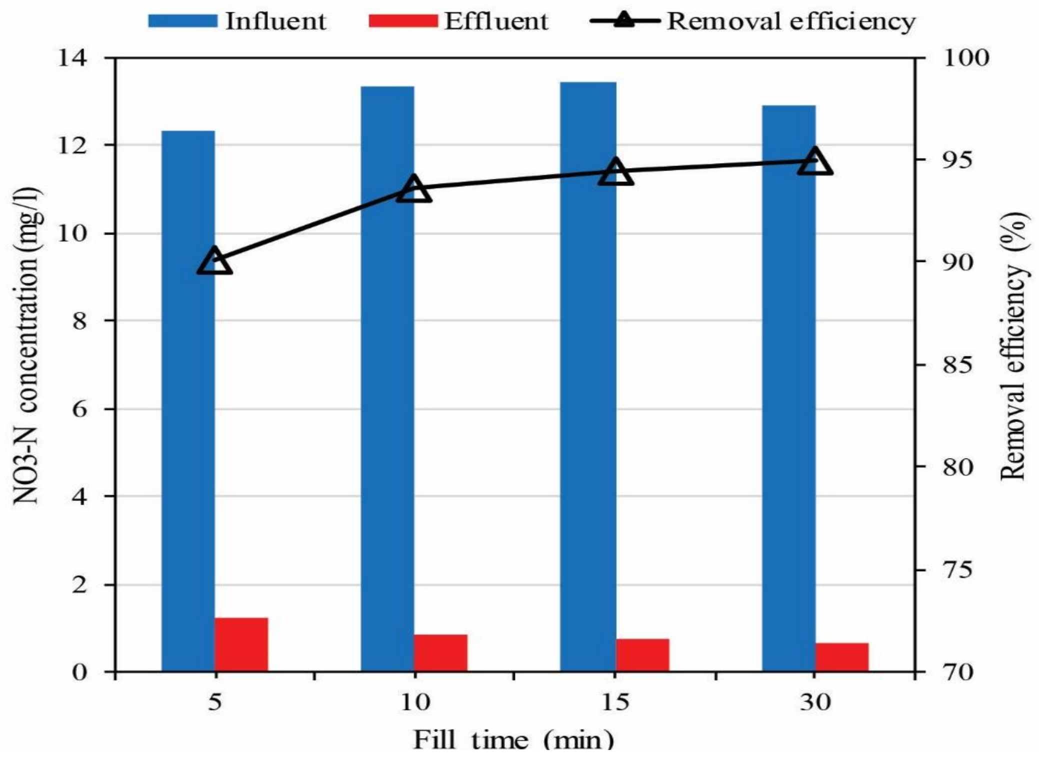



Figure 5.22: The effect of fill time on $NO_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs*

### 5.1.4.4 The Effect of fill time on $NO_2$-N removal

The effect of fill time on $NO_2$-N removal efficiency is shown in Figure 5.23. The removal efficiency of $NO_2$-N at 5 minutes fill time was 95.1%. It can be seen from the results that the $NO_2$-N removal efficiency has improved when the fill time increased from 5 minutes to 10 minutes; it was raised from 95.1% to 96.8%. This may be because longer fill time gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates (Thakur et al., 2013b). However, when the fill time increased to 15 minutes, there was no significant improve in the removal efficiency of $NO_2$-N; it was 96.1% at 15 minutes fill time. Moreover, the $NO_2$-N removal efficiency was not affected when the fill time increased from 15 minutes to 30 minutes; it was 96.5% at 30 minutes fill time.

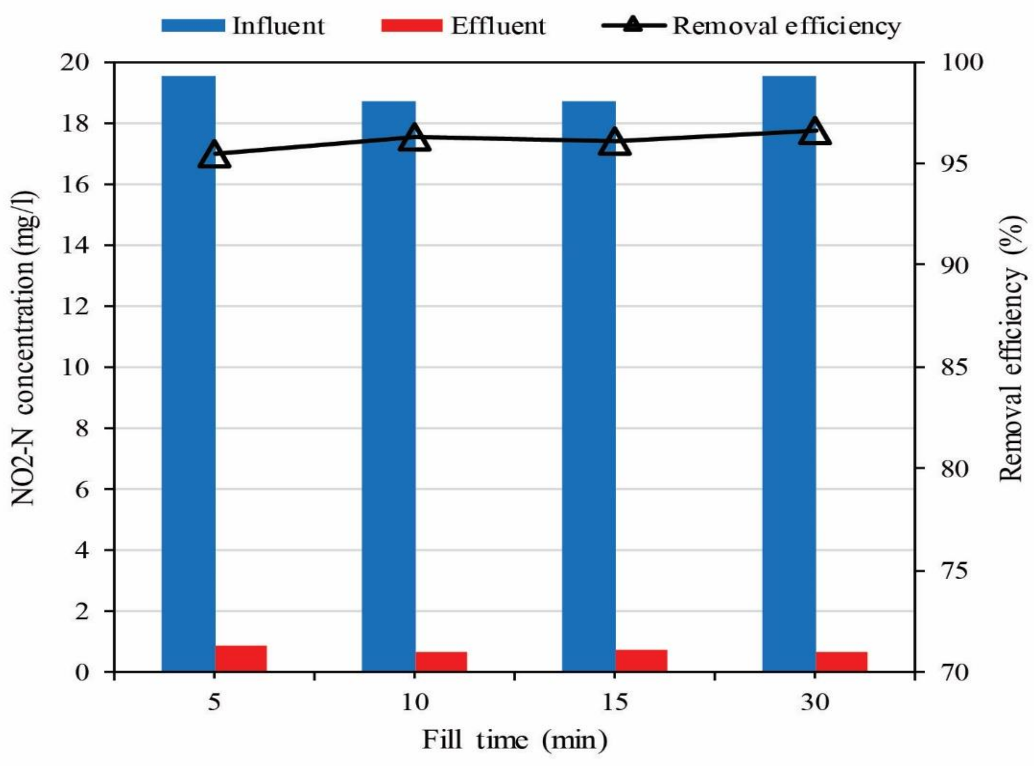



Figure 5.23: The effect of fill time on $NO_2$-N removal

*<u>MLSS 4 g/l; HRT 5.5 hrs</u>*

#### 5.1.4.5 The Effect of fill time on sludge settleability

Figure 5.24 shows the SVI values under different fill times. SVI values were 31.9 ml/g, 32 ml/g, 33.5 ml/g and 32.9 ml/g for fill time of 5 minutes, 10 minutes, 15 minutes and 30 minutes respectively. The results showed that increasing the fill time from 5 to 10, 15 and 30 minutes had no significant effect on the sludge settleability, while Thakur et al. (2013b) stated that settling performance improved when the feeding time increased to 2 hours. Although there was no significant improvement when the fill time increased from 5 to 10, 15 and 30 minutes, all of the treatment reactors had a very good settling performance.

The fill time results obtained from this study are in agreement with Damasceno et al. (2007) who reported that longer feeding time is better for a biodegrading high concentration of COD. However, Thakur et al. (2013b) achieved negative results when increasing the feeding time. While Sahinkaya and Dilek (2007) found no effect on feeding time on the nutrient removal efficiency. This study suggests that 30 minutes fill time is the optimal range for the TSSBR system operation.

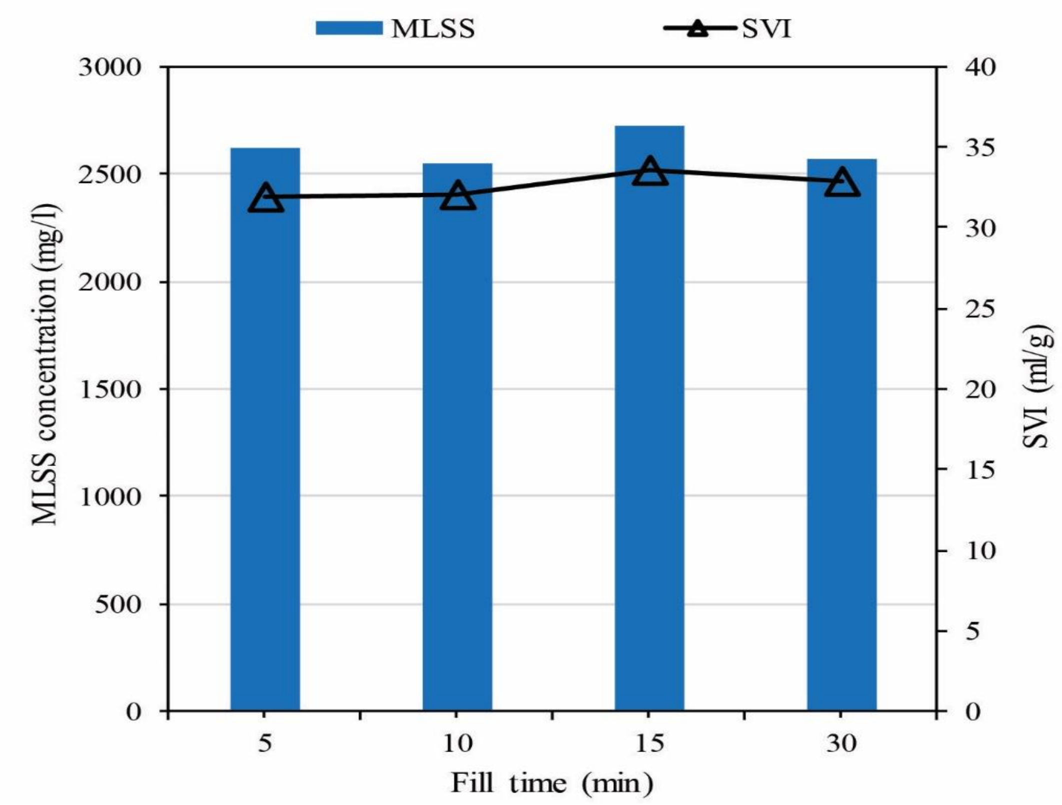



Figure 5.24: The effect of fill time on Sludge characteristics
*MLSS 4 g/l; HRT 5.5 hrs*

### 5.1.5 **Volumetric exchange rate**

The ratio of the influent wastewater volume that enters the treatment reactor to the reactor's working volume is called volumetric exchange rate (VER) (Tsang et al., 2007). It reflects the treatment capacity of a single SBR operation cycle. In this research, the four treatment reactors operated under four volumetric exchange rates (20%, 40%, 60% and 70%). The effects of VER on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance test) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.5.1 **The Effect of VER on COD removal**

The effect of VER on COD removal efficiency is shown in Figure 5.25. The removal efficiency of COD at 20% VER was 94.4% when the VER increased to 40%; there was no significant change in the removal efficiency of COD, it was 93.7% at 40% VER. However, it can be seen from the results that the COD removal efficiency has significantly declined when the VER increased from 40% to 60%; it decreased from 93.7% to 89.6%. Moreover, the COD removal efficiency declined furthermore when the VER increased from 60% to 70%; it was 84.4% at 70% VER. This agreed with Tsang et al. (2007), who stated that high VER results in poor effluent quality.

Arnz et al. (2000) studied simultaneous loading and draining as a means to enhance the efficiency of sequencing biofilm batch reactors (SBBR), they stated that at a volumetric exchange rate of 68% in the lab-scale SBBR and 90% in the semi-full-scale system, respectively, the removal rates were significant.

Zielinska et al. (2012) studied nitrogen removal from wastewater and bacterial diversity in the activated sludge at different COD/N ratios and dissolved oxygen concentrations. He reached up to 93% of COD removal with 50% volumetric exchange rate.

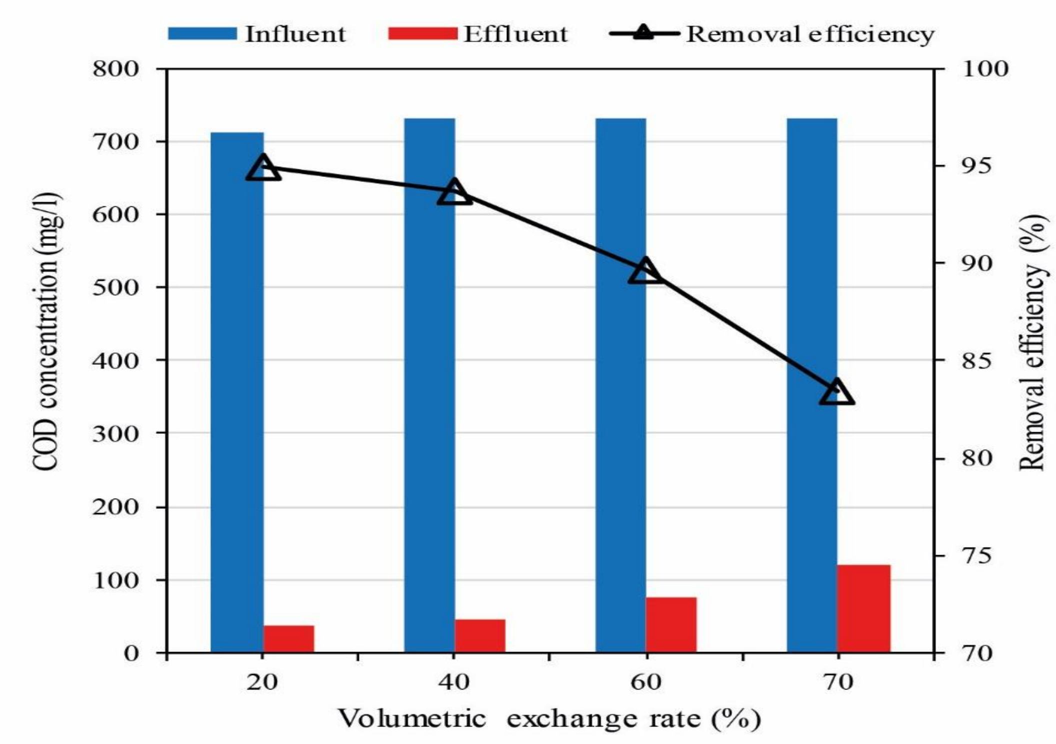



Figure 5.25: The effect of VER on COD removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.5.2 The Effect of VER on $NH_3$-N removal

The effect of VER on $NH_3$-N removal efficiency is shown in Figure 5.26. The removal efficiency of $NH_3$-N at 20% VER was 95.3% when the VER increased to 40%; there was no significant change in the removal efficiency of $NH_3$-N, it was 95.1% at 40% VER. However, it can be seen from the results that the $NH_3$-N removal efficiency has significantly declined when the VER increased from 40% to 60%; it decreased from 95.1% to 90.7%. Moreover, the $NH_3$-N removal efficiency declined furthermore when the VER increased from 60% to 70%; it was 84.6% at 70% VER. This agreed with Tsang et al. (2007), who stated that high VER results in poor effluent quality. Zielinska et al. (2012) stated that with 50% volumetric exchange rate, the ammonia concentration in the effluent did not exceed 0.5 mg/l.

Bernat et al. (2011) studied the removal of nitrogen from wastewater with a low COD/N ratio at a low oxygen concentration, during the experiment; three series differing in the volumetric exchange rate (10%, 30% and 50%) were conducted. At a volumetric exchange rate of 10% and 30%, total ammonia was removed in the first aeration phase. At the highest volumetric exchange rate of 50%, a significant increase in the ammonia nitrogen concentration at the beginning of the SBR cycle to about 90 mg $NH_3$-N/L resulted in only about 80% of ammonia nitrogen being oxidized in the first aeration phase. Complete oxidation occurred in the second aeration phase.

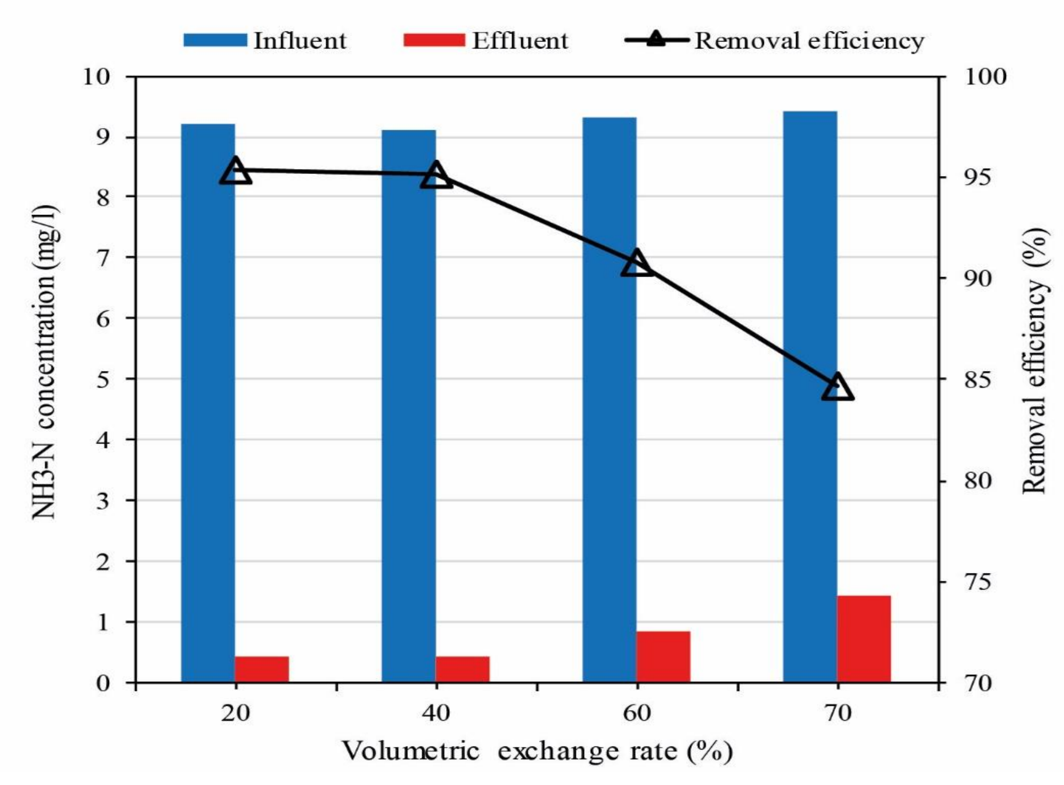



Figure 5.26: The effect of VER on $NH_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.5.3 **The Effect of VER on $NO_3$-N removal**

The effect of VER on $NO_3$-N removal efficiency is shown in Figure 5.27. The removal efficiency of $NO_3$-N at 20% VER was 93.3% when the VER increased to 40%; there was no significant change in the removal efficiency of $NO_3$-N, it was 92.9% at 40% VER. However, it can be seen from the results that the $NO_3$-N removal efficiency has significantly declined when the VER increased from 40% to 60%; it was decreased from 92.9% to 89.6%. Moreover, the $NO_3$-N removal efficiency was declined furthermore when the VER increased from 60% to 70%; it was 83.1% at 70% VER. This agreed with Tsang et al. (2007), who stated that high VER results in poor effluent quality. Zielinska et al. (2012) stated that with 50% volumetric exchange rate, the effluent nitrate concentration was 33.2 mg/l.

Bernat et al. (2011) used three series of volumetric exchange rate (10%, 30% and 50%), when studying the removal of nitrogen from wastewater with a low COD/N ratio at a low oxygen concentration, stated that at the low volumetric exchange rate the main product of ammonia nitrogen oxidation was nitrates – no accumulation of nitrite in the SBR cycle was observed. When increasing the volumetric exchange rate to 30% resulted in the appearance of nitrite, its concentration in the effluent did not exceed 0.1 mg/l. Finally, at the volumetric exchange rate of 50%, small amounts of nitrites grew during ammonia nitrogen oxidation, but the final nitrification product was nitrate. The nitrate concentration in the effluent at the volumetric exchange rates of 10%, 30% and 50% was about 110, 130 and 85 mg/l, respectively.

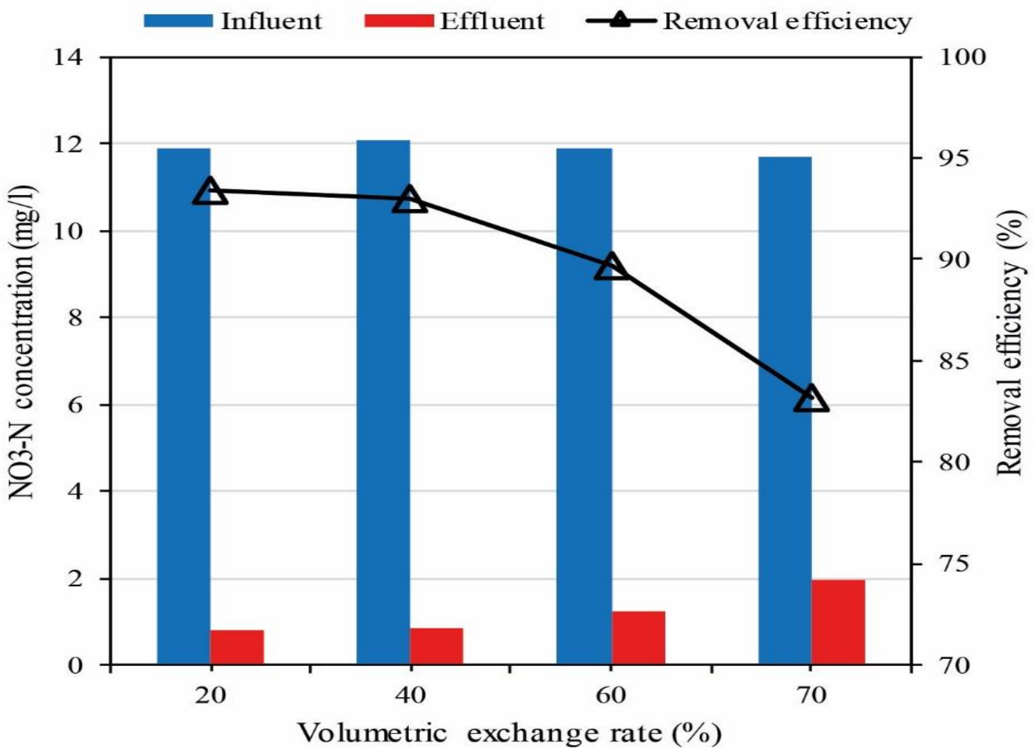



Figure 5.27: The effect of VER on $NO_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.5.4 The Effect of VER on $NO_2$-N removal

The effect of VER on $NO_2$-N removal efficiency is shown in Figure 5.28. The removal efficiency of $NO_2$-N at 20% VER was 95.6% when the VER increased to 40%; there was no significant change in the removal efficiency of $NO_2$-N, it was 95.1% at 40% VER. However, it can be seen from the results that the $NO_2$-N removal efficiency has significantly declined when the VER increased from 40% to 60%; it decreased from 95.1% to 92%. Moreover, the $NO_2$-N removal efficiency declined furthermore when the VER increased from 60% to 70%; it was 86.5% at 70% VER. This agreed with Tsang et al. (2007), who stated that high VER results in poor effluent quality. Zielinska et al. (2012) stated that with 50% volumetric exchange rate, the effluent nitrite concentration did not exceed 0.03 mg/l.

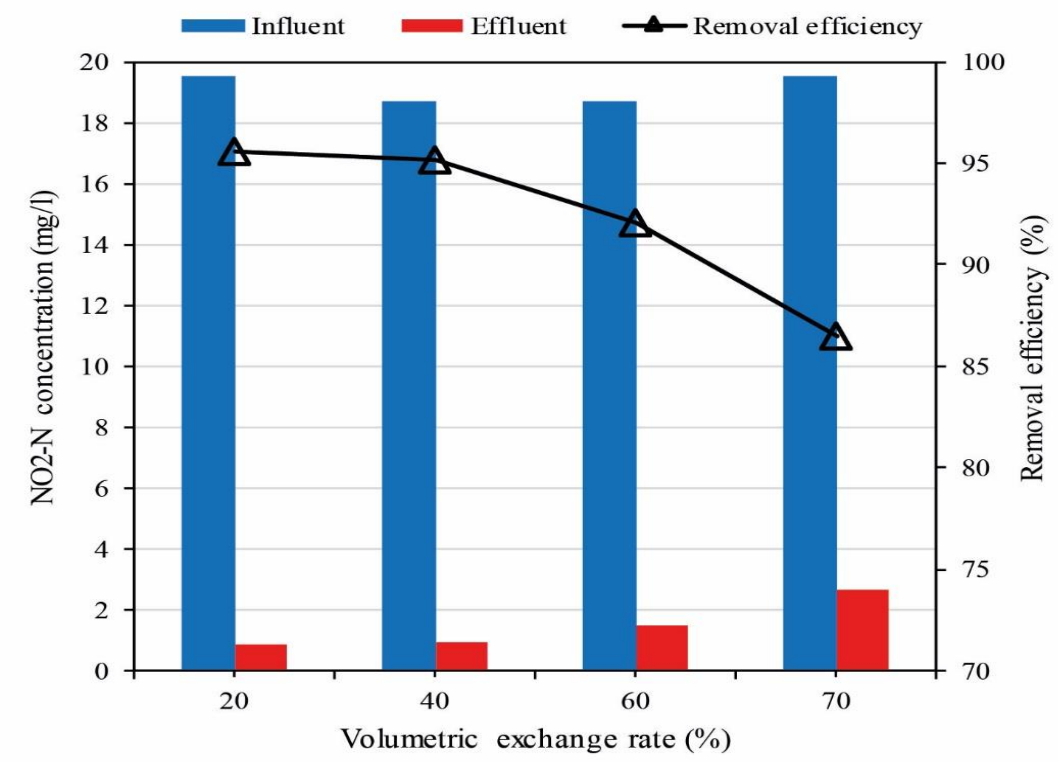



Figure 5.28: The effect of VER on $NO_2$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.5.5 **The Effect of VER on sludge settleability**

The effect of VER on sludge settleability is shown in Figure 5.29. The SVI value at 20% VER was 44.2 ml/g when the VER increased to 40%; the SVI value declined to 42.1 ml/g. In addition, it can be seen from the results that the SVI value has significantly declined when the VER increased from 40% to 60%; it decreased from 42.1 ml/g to 35.5 ml/g. Moreover, the SVI value declined furthermore when the VER increased from 60% to 70%; it was 34.7% at 70% VER. This might be due to the significant gap of the organic substrate produced between before and after feed-filling in the reactor, meaning that high VER value is usually regarded as an advantage for preventing sludge bulking (Martins et al., 2003). Li et al. (2017) stated that the microbial community structure changed at the high volumetric exchange rate, which had a negative impact on settling performance.

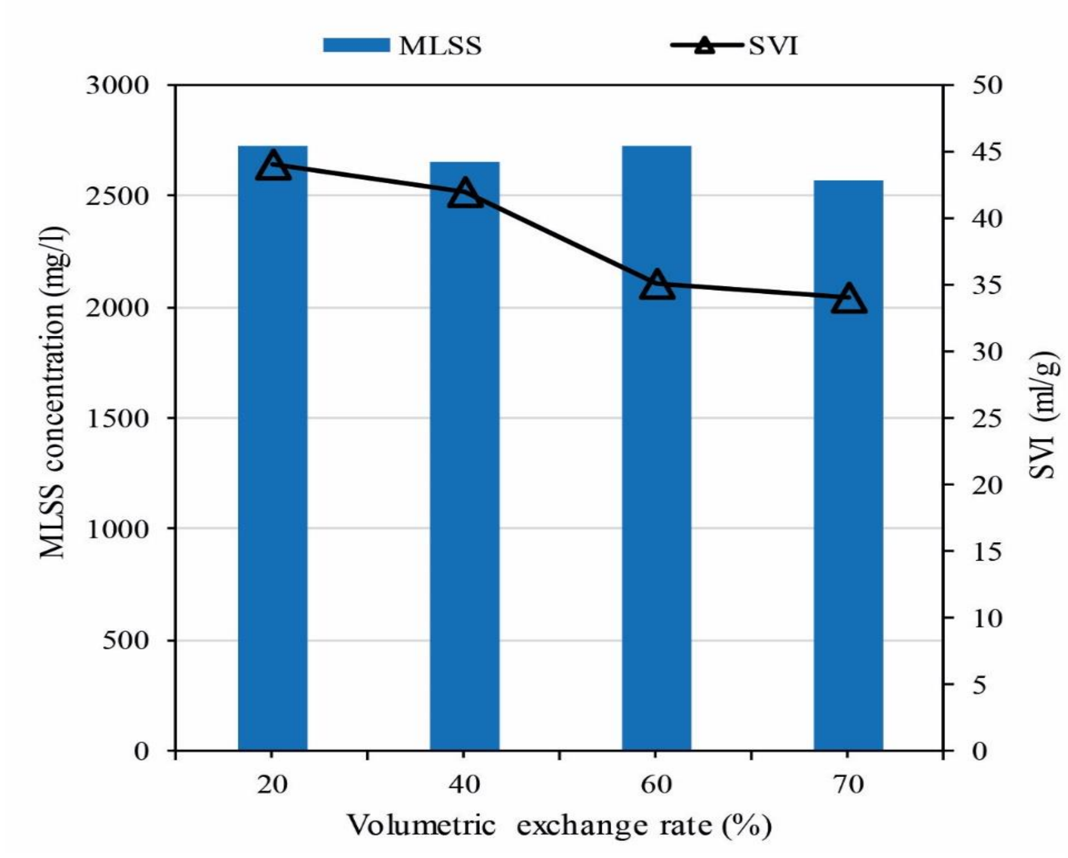



Figure 5.29: The effect of VER on sludge characteristics
*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.6 **Organic loading rate (Glucose)**

The four treatment reactors were operated at different glucose concentrations (750, 1000, 1250 and 1500 mg/l). The effects of OLR on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance test) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.6.1 **The Effect of glucose loading rate on COD removal**

The effect of glucose loading rate on COD removal efficiency is shown in Figure 5.30. The removal efficiency of COD at a glucose concentration of 750 mg/l was 93.2% when the glucose concentration increased to 1000 mg/l; there was no significant change in the removal efficiency of COD, it was 92.9% at 1000 mg glucose/l. In addition, increasing the glucose concentration from 1000 mg/l to 1250 mg/l had no significant effect on COD removal efficiency; it was 93% at 1250 mg glucose/l. However, the COD removal efficiency was significantly reduced when the glucose concentration increased from 1250 mg/l to 1500 mg/l; it decreased from 93% to 89.8%. This result agreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD, and nitrogen compounds removal rates even under high ORL.

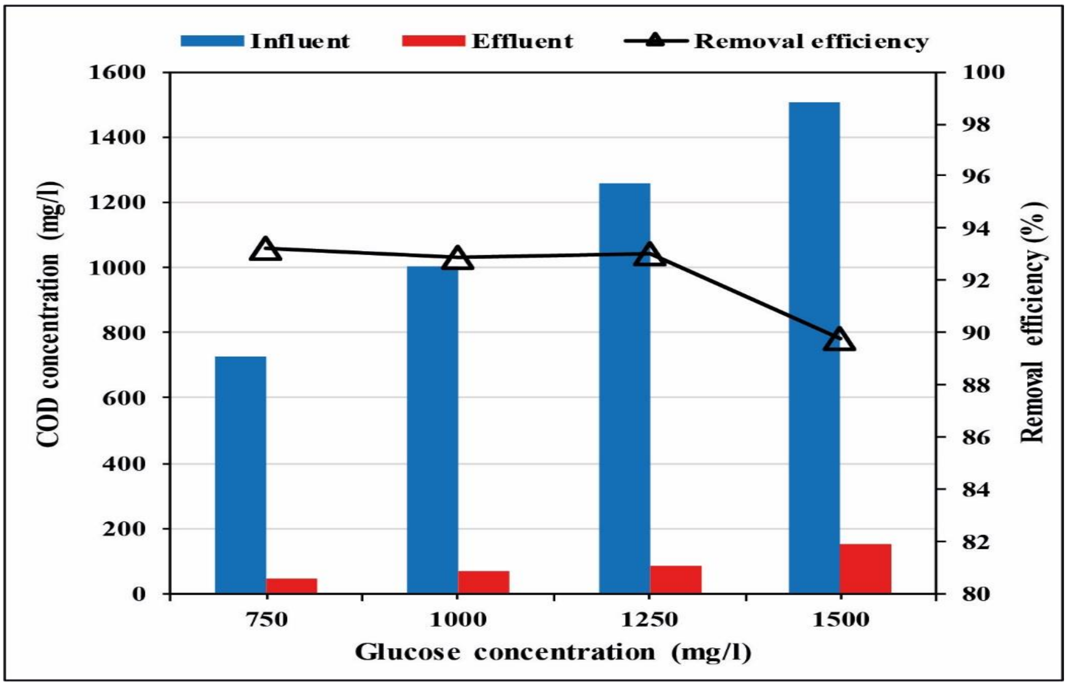



Figure 5.30: The effect of glucose loading rate on COD removal
Source (Alattabi et al., 2017b)

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.6.2 The Effect of glucose loading rate on $NH_3$-N removal

The effect of glucose loading rate on $NH_3$-N removal efficiency is shown in Figure 5.31. The removal efficiency of $NH_3$-N at a glucose concentration of 750 mg/l was 95.2%, when the glucose concentration increased to 1000 mg/l, there was no significant change in the removal efficiency of $NH_3$-N, it was 95.9% at 1000 mg glucose/l. In addition, increasing the glucose concentration from 1000 mg/l to 1250 mg/l had no significant effect on $NH_3$-N removal efficiency; it was 94.6% at 1250 mg glucose/l. However, the $NH_3$-N removal efficiency was significantly reduced when the glucose concentration increased from 1250 mg/l to 1500 mg/l; it decreased from 94.6% to 91%. This result agreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

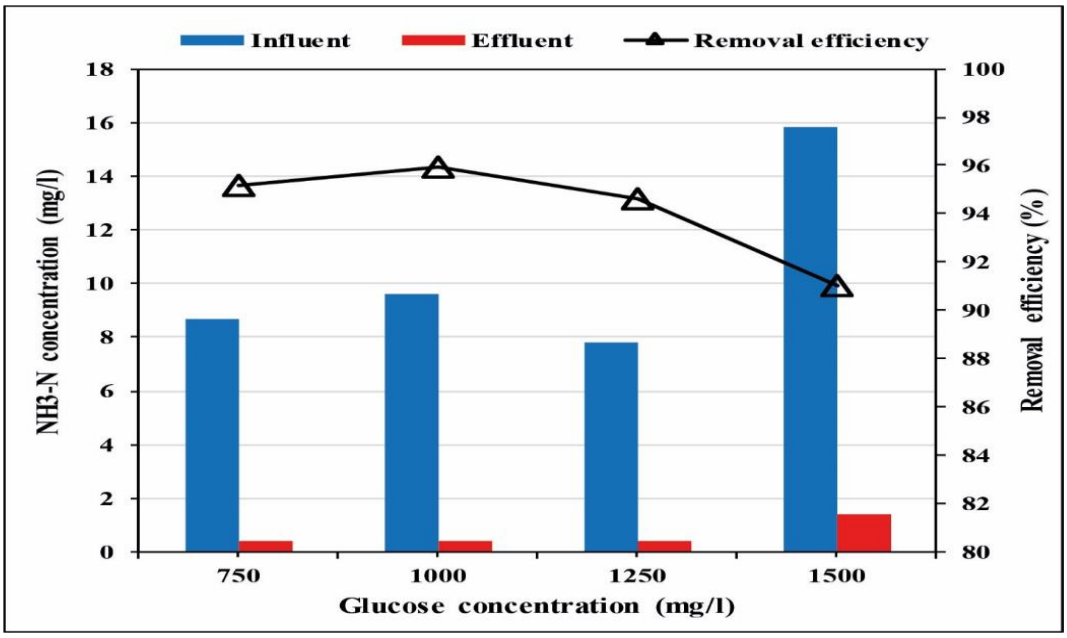



Figure 5.31: The effect of glucose loading rate on $NH_3$-N removal
Source (Alattabi et al., 2017b)

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.6.3 **The Effect of glucose loading rate on $NO_3$-N removal**

The effect of glucose loading rate on $NO_3$-N removal efficiency is shown in Figure 5.32. The removal efficiency of $NO_3$-N at a glucose concentration of 750 mg/l was 94.9%, when the glucose concentration increased to 1000 mg/l, there was no significant change in the removal efficiency of $NO_3$-N, it was 93.6% at 1000 mg glucose/l. In addition, increasing the glucose concentration from 1000 mg/l to 1250 mg/l had no significant effect on $NO_3$-N removal efficiency; it was 94.1% at 1250 mg glucose/l. However, the $NO_3$-N removal efficiency was significantly reduced when the glucose concentration increased from 1250 mg/l to 1500 mg/l; it decreased from 94.1% to 88.8%. This result agreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

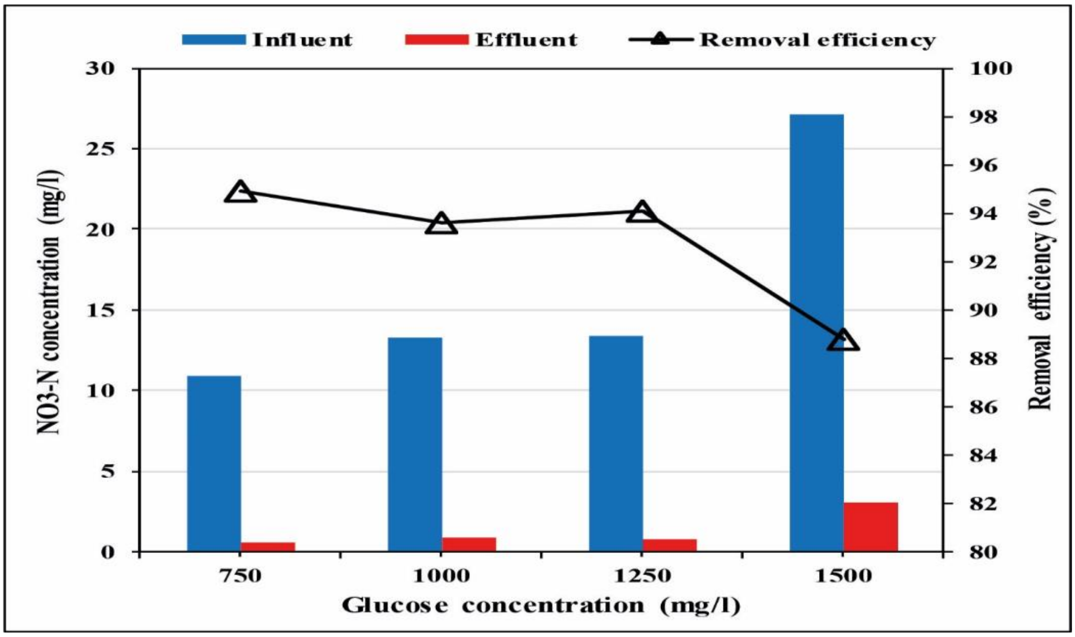



Figure 5.32: The effect of glucose loading rate on $NO_3$-N removal

Source (Alattabi et al., 2017b)

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.6.4 **The Effect of glucose loading rate on $NO_2$-N removal**

The effect of glucose loading rate on $NO_2$-N removal efficiency is shown in Figure 5.33. The removal efficiency of $NO_2$-N at a glucose concentration of 750 mg/l was 96.5%, when the glucose concentration increased to 1000 mg/l, there was no significant change in the removal efficiency of $NO_2$-N, it was 95.2% at 1000 mg glucose/l. In addition, increasing the glucose concentration from 1000 mg/l to 1250 mg/l had no significant effect on $NO_2$-N removal efficiency; it was 96.1% at 1250 mg glucose/l. However, the $NO_2$-N removal efficiency was significantly reduced when the glucose concentration increased from 1250 mg/l to 1500 mg/l; it decreased from 96.1% to 92%. This result agreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

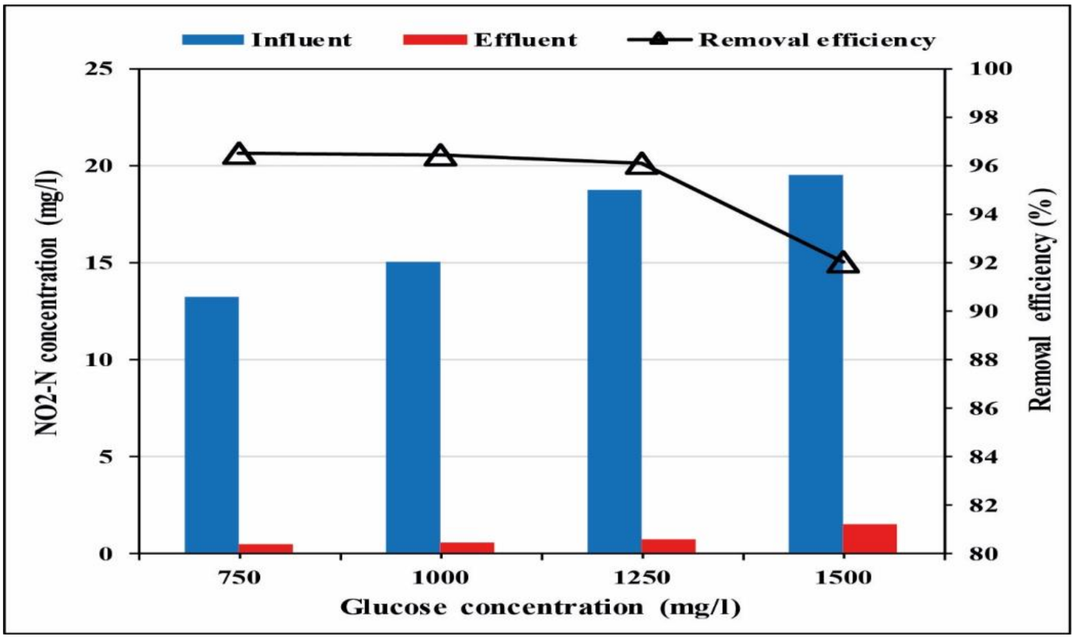



Figure 5.33: The effect of glucose loading rate on $NO_2$-N removal
Source (Alattabi et al., 2017b)
*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.6.5 The Effect of glucose loading rate on sludge settleability

The effect of glucose loading rate on the sludge settleability is shown in Figure 5.34. The SVI value at a glucose concentration of 750 mg/l was 32.1 ml/g when the glucose concentration increased to 1000 mg/l; the SVI value was increased to 34.6 ml/g. In addition, increasing the glucose concentration from 1000 mg/l to 1250 mg/l led to increasing the SVI value from 34.6 ml/g to 39.2 ml/g. Moreover, the SVI value was significantly raised when the glucose concentration increased from 1250 mg/l to 1500 mg/l; it was increased from 39.2 ml/g to 42.7 ml/g. In the same vein, Xu et al. (2014) stated that increasing the OLR will lead to a proportional increase in biomass concentration, which will result in high SVI and the settleability of the solids will decrease. This agreed with Bassin et al. (2016), who reported an increase in the concentration of suspended solids when the initial concentration of COD was increased, which would also lead to an increase in the SVI and a subsequent drop in the solids' settleability. However, the results obtained disagreed with (Chan and Lim, 2007) who evaluated a SBR performance with aerated and un-aerated fill periods in treating phenol-containing wastewater, they reported that a serious bulking problem was recorded for lower influent phenol concentrations at 300 and 500 mg/L as the growth of filamentous bacteria was not suppressed due to their higher surface area to volume ratio which enables them to obtain food and store the excessive nutrient in a comparatively more efficient manner. The difference in the results might be because of the difference in the laboratory setup of the treatment system

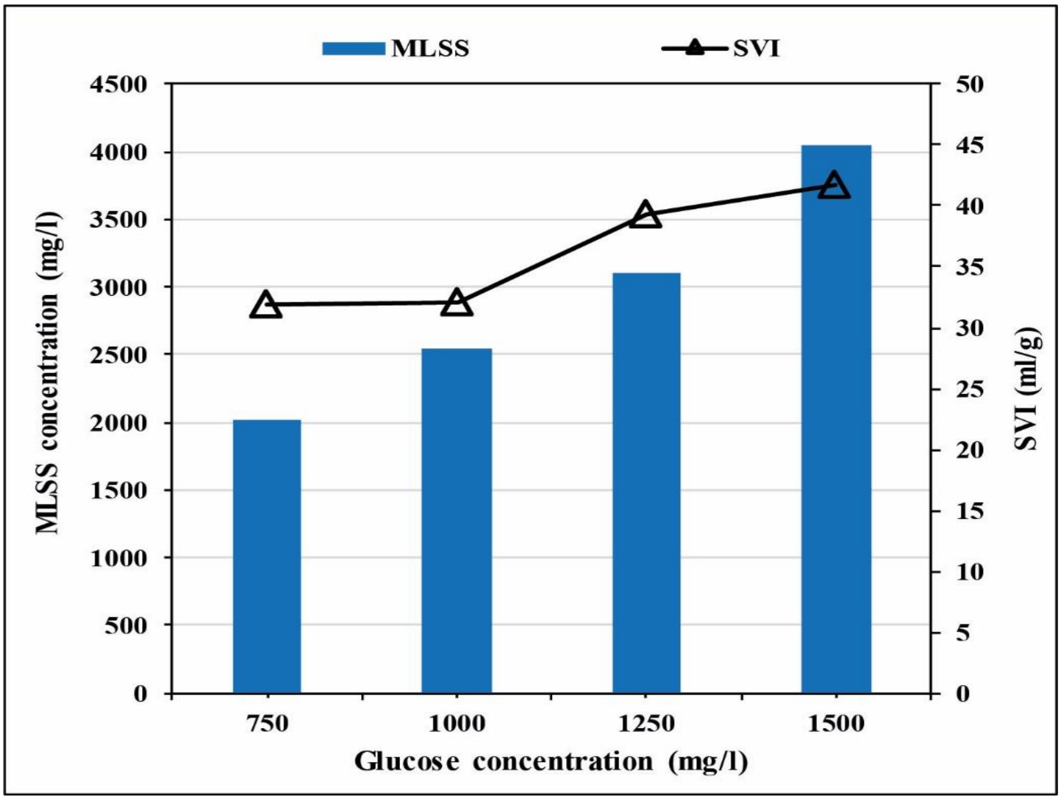



Figure 5.34: The effect of glucose loading rate on sludge settleability

Source (Alattabi et al., 2017b)

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.7 **Organic loading rate (Potassium-nitrate)**

The four treatment reactors were operated under different potassium-nitrate concentrations (50, 100, 150 and 200 mg/l). The effects of OLR on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance test) in an attempt to improve settling performance and enhance effluent quality.

#### 5.1.7.1 **The Effect of potassium-nitrate loading rate on COD removal**

The effect of potassium-nitrate loading rate on COD removal efficiency is shown in Figure 5.35. The removal efficiency of COD at a potassium-nitrate concentration of 50 mg/l was 91.7% when the potassium-nitrate concentration increased to 100 mg/l;

there was no significant change in the removal efficiency of COD, it was 91.6% at 100 mg potassium-nitrate/l. In addition, increasing the potassium-nitrate concentration from 100 mg/l to 150 mg/l had no significant effect on COD removal efficiency; it was 90.8% at 150 mg potassium-nitrate/l. Moreover, the COD removal efficiency was not affected when the potassium-nitrate concentration increased from 150 mg/l to 200 mg/l; it was 91.9% at 200 mg potassium-nitrate/l. This result disagreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

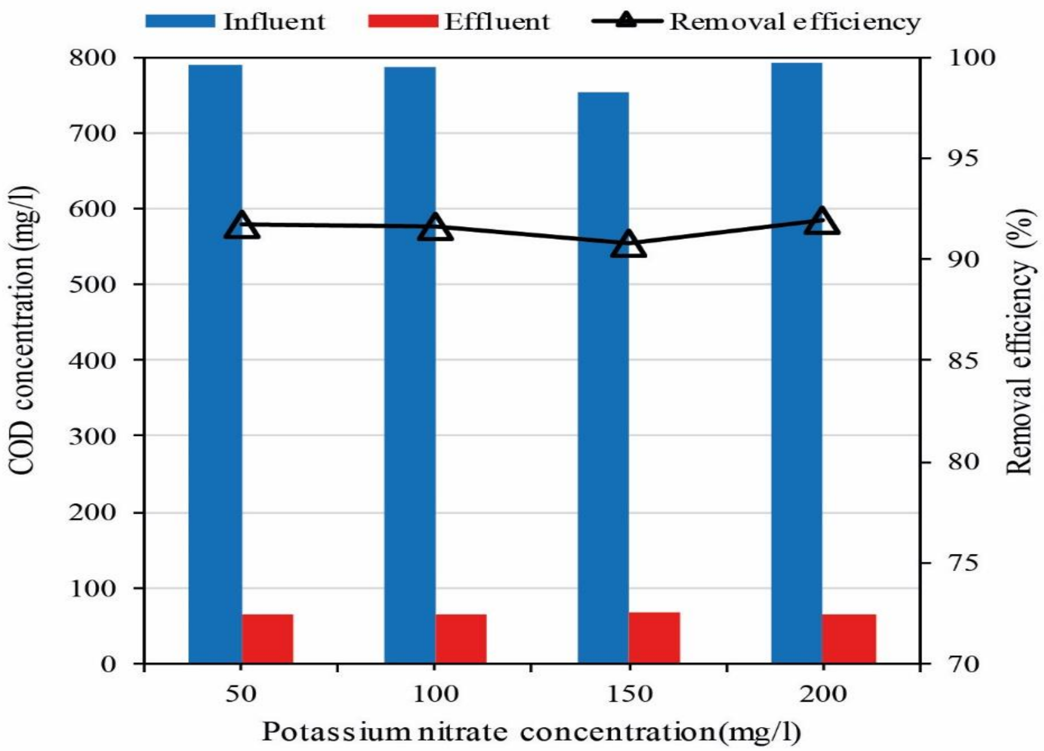



Figure 5.35: The effect of potassium-nitrate loading rate on COD removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.7.2 **The Effect of potassium-nitrate loading rate on $NH_3$-N removal**

The effect of potassium-nitrate loading rate on $NH_3$-N removal efficiency is shown in Figure 5.36. The removal efficiency of $NH_3$-N at a potassium-nitrate concentration of 50 mg/l was 91.3% when the potassium-nitrate concentration increased to 100 mg/l; there was no significant change in the removal efficiency of $NH_3$-N, it was 91.5% at 100 mg potassium-nitrate/l. In addition, increasing the potassium-nitrate concentration from 100 mg/l to 150 mg/l had no significant effect on $NH_3$-N removal efficiency; it was 91.1% at 150 mg potassium-nitrate/l. However, the $NH_3$-N removal efficiency significantly declined when the potassium-nitrate concentration increased from 150 mg/l to 200 mg/l; it was decreased from 91.1% to 86.7%. This result agreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

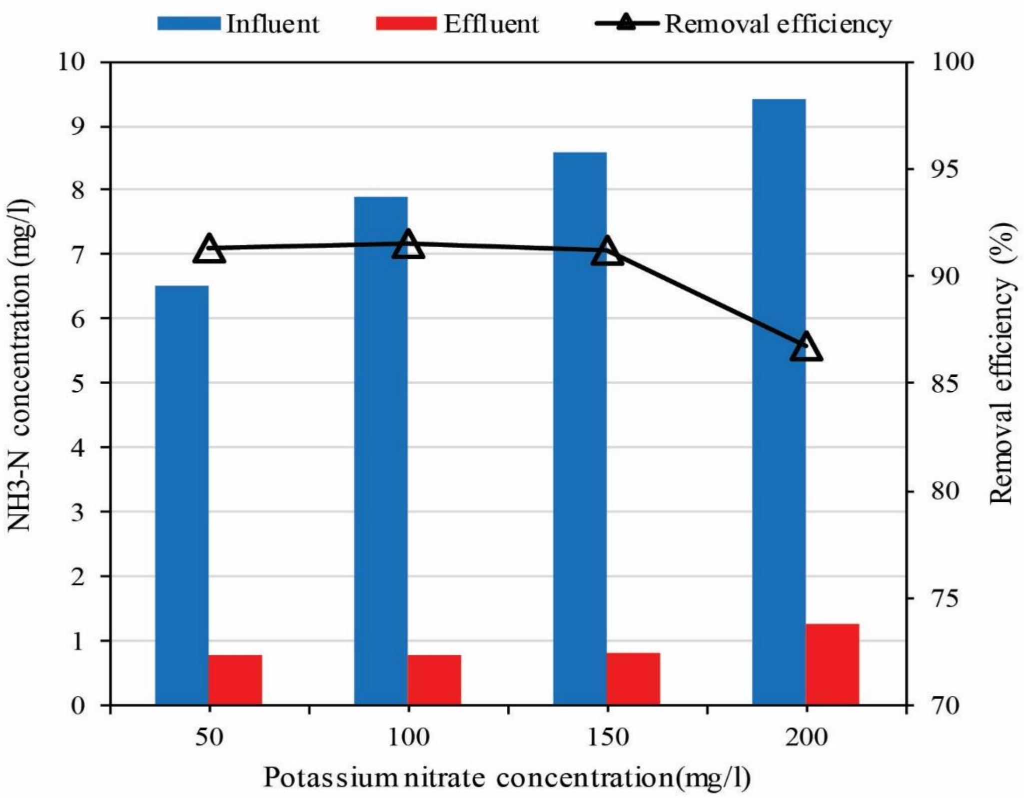



Figure 5.36: The effect of potassium-nitrate loading rate on $NH_3$-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.7.3 **The Effect of potassium-nitrate loading rate on $NO_3$-N removal**

The effect of potassium-nitrate loading rate on $NO_3$-N removal efficiency is shown in Figure 5.37. The removal efficiency of $NO_3$-N at a potassium-nitrate concentration of 50 mg/l was 92.2% when the potassium-nitrate concentration increased to 100 mg/l; there was no significant change in the removal efficiency of $NO_3$-N, it was 91.7% at 100 mg potassium-nitrate/l. In addition, increasing the potassium-nitrate concentration from 100 mg/l to 150 mg/l had no significant effect on $NO_3$-N removal efficiency; it was 91.9% at 150 mg potassium-nitrate/l. However, the $NO_3$-N removal efficiency significantly declined when the potassium-nitrate concentration increased from 150 mg/l to 200 mg/l; it decreased from 91.9% to 87.1%. This result agreed with (Liu and

Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

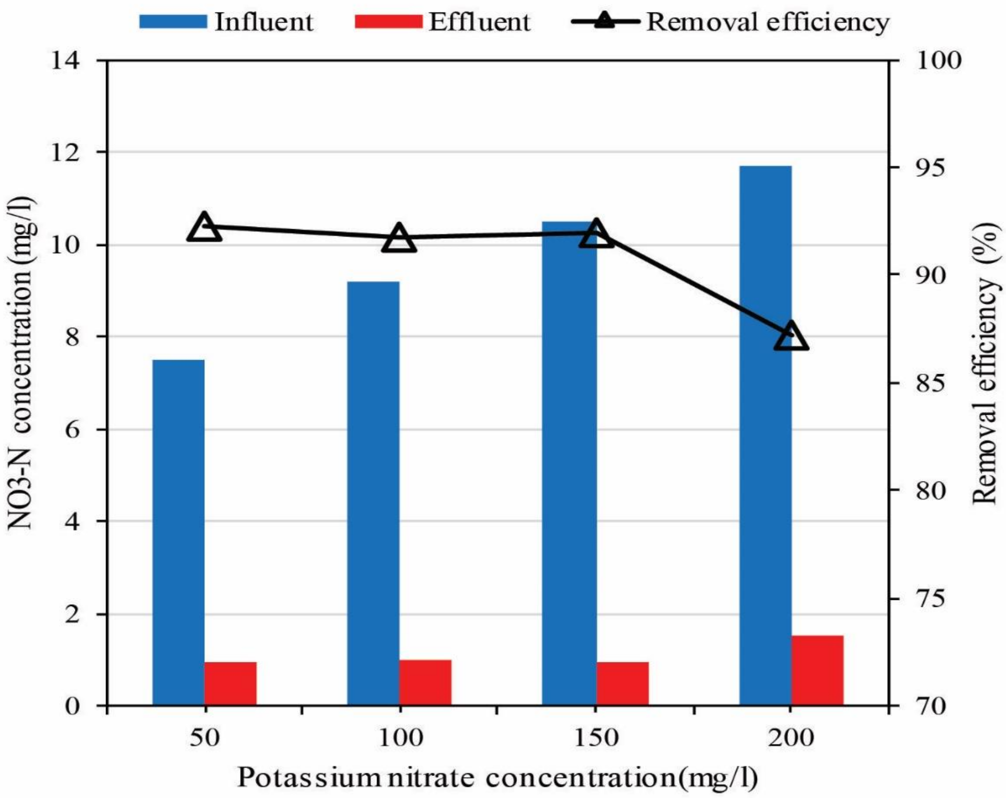



Figure 5.37: The effect of potassium-nitrate loading rate on $NO_3$-N removal
*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

#### 5.1.7.4 **The Effect of potassium-nitrate loading rate on $NO_2$-N removal**

The effect of potassium-nitrate loading rate on $NO_2$-N removal efficiency is shown in Figure 5.38. The removal efficiency of $NO_2$-N at a potassium-nitrate concentration of 50 mg/l was 92.8% when the potassium-nitrate concentration increased to 100 mg/l; there was no significant change in the removal efficiency of $NO_2$-N, it was 92.1% at 100 mg potassium-nitrate/l. In addition, increasing the potassium-nitrate concentration from 100 mg/l to 150 mg/l had no significant effect on $NO_2$-N removal efficiency; it was 91.9% at 150 mg potassium-nitrate/l. Moreover, the $NO_2$-N removal efficiency was not affected when the potassium-nitrate concentration increased from 150 mg/l to 200 mg/l; it was 91.2% at 200 mg potassium-nitrate/l. This result disagreed with (Liu and Tay, 2004), who stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

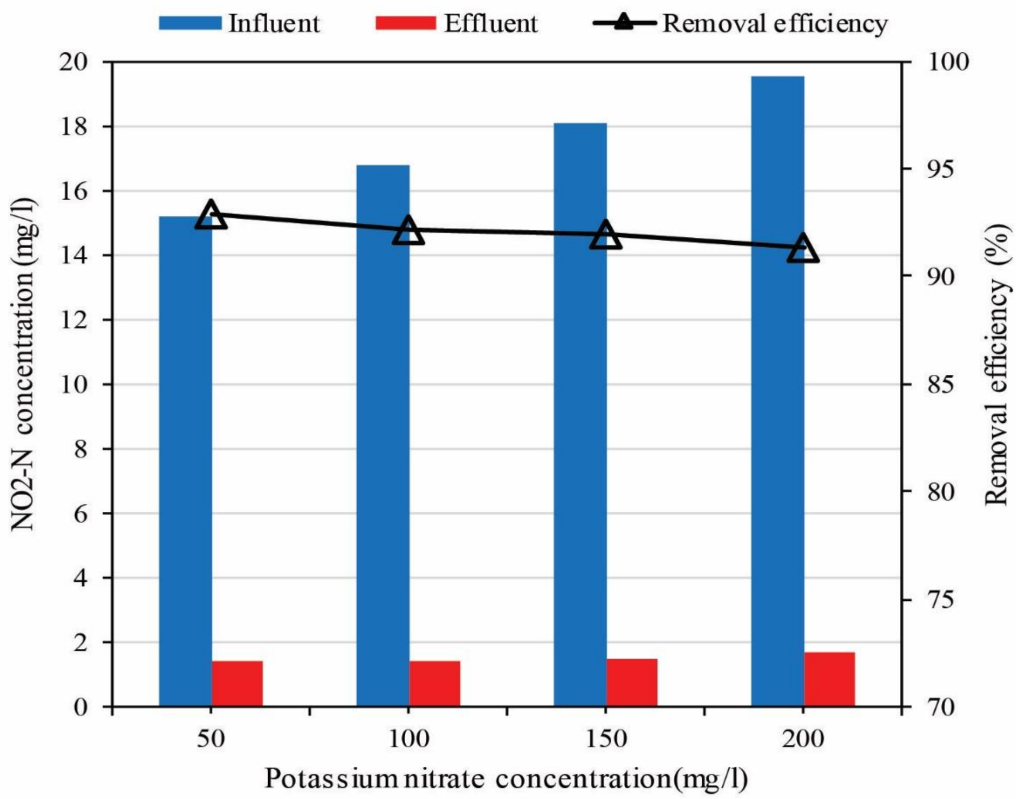



Figure 5.38: The effect of potassium-nitrate loading rate on NO2-N removal

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.7.5 **The Effect of potassium-nitrate loading rate on sludge settleability**

The effect of potassium-nitrate loading rate on the sludge settleability is shown in Figure 5.39. The SVI value at a potassium-nitrate concentration of 50 mg/l was 32.3 ml/g when the potassium-nitrate concentration increased to 100 mg/l; there was no significant change in the SVI value; it was 33.1 ml/g. In addition, increasing the potassium-nitrate concentration from 100 mg/l to 150 mg/l led to increasing the SVI value from 33.1 ml/g to 37 ml/g. Moreover, the SVI value was significantly raised when the potassium-nitrate concentration increased from 150 mg/l to 200 mg/l; it increased from 37 ml/g to 40.1 ml/g. In the same vein, Xu et al. (2014) stated that increasing the OLR will lead to a proportional increase in biomass concentration, which will result in high SVI and the settleability of the solids will decrease. This agreed with Bassin et al. (2016), who reported an increase in the concentration of suspended solids when the initial concentration of COD was increased, which would also lead to an increase in the SVI and a subsequent drop in the solids' settleability.

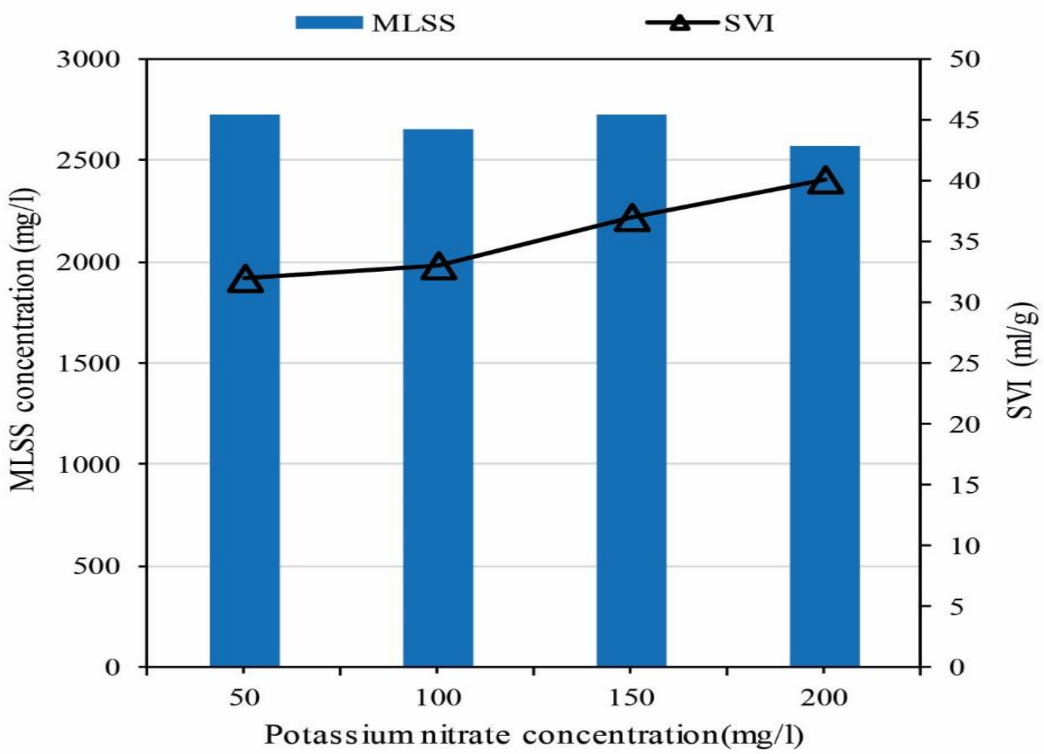



Figure 5.39: The effect of potassium-nitrate loading rate on sludge settleability

*MLSS 4 g/l; HRT 5.5 hrs; Fill 15 min*

### 5.1.8 Hydraulic shock

Two hydraulic shocks have been applied to the TSSBR system by decreasing the cycle time into half cycle time and then 3/8 cycle time, the system has operated under these shocks for one treatment cycle and then two treatment cycles. The effects of hydraulic shock on both settleability and effluent quality in a TSSBR was investigated through a series of experiments (water quality parameters' removal efficiency and settling performance test) in an attempt to improve settling performance and enhance effluent quality.

The TSSBR system was normally operated until it reached the steady-state operation. Then hydraulic shocks were created by decreasing the cycle time (5.5 h) of the reactor.

The hydraulic shocks were applied for both single-cycle shock (SCS) and double-cycle shock (DCS). The operating sequence of TSSBR during hydraulic shocks is shown in Table 5-2. During the first stage, the cycle time was reduced to half of the normal value, which was approximately 2.75 h. In the subsequent stage, the cycle time was reduced to 2 h, which was 3/8 of the initial normal value.

Table 5-2: TSSBR operating conditions during hydraulic shocks

| Operating condition | Cycle time (h) | Number of cycles |
|---|---|---|
| Steady state operation | 5.5 | 9 |
| Hydraulic shock (1/2) cycle (SCS) | 2.75 | 1 |
| Normal condition | 5.5 | 9 |
| Hydraulic shock (1/2) cycle (DCS) | 2.75 | 2 |
| Normal condition | 5.5 | 9 |
| Hydraulic shock (3/8) cycle (SCS) | 2 | 1 |
| Normal condition | 5.5 | 9 |
| Hydraulic shock (3/8) cycle (DCS) | 2 | 2 |

#### 5.1.8.1 The Effect of hydraulic shock on COD removal

The removal efficiency of COD during the hydraulic shocks is shown in Figure 5.40. During the steady-state operation, the removal efficiency of COD for SCS and DCS was 88.9% and 89.7% respectively. After the first shock (1/2 cycle), the removal efficiency of COD for SCS and DCS dropped to 83.6% and 79.2% respectively. In addition, the removal efficiency of COD for SCS and DCS declined to 81.2% and 76.1% respectively, when the second shock (3/8 cycle) was applied.

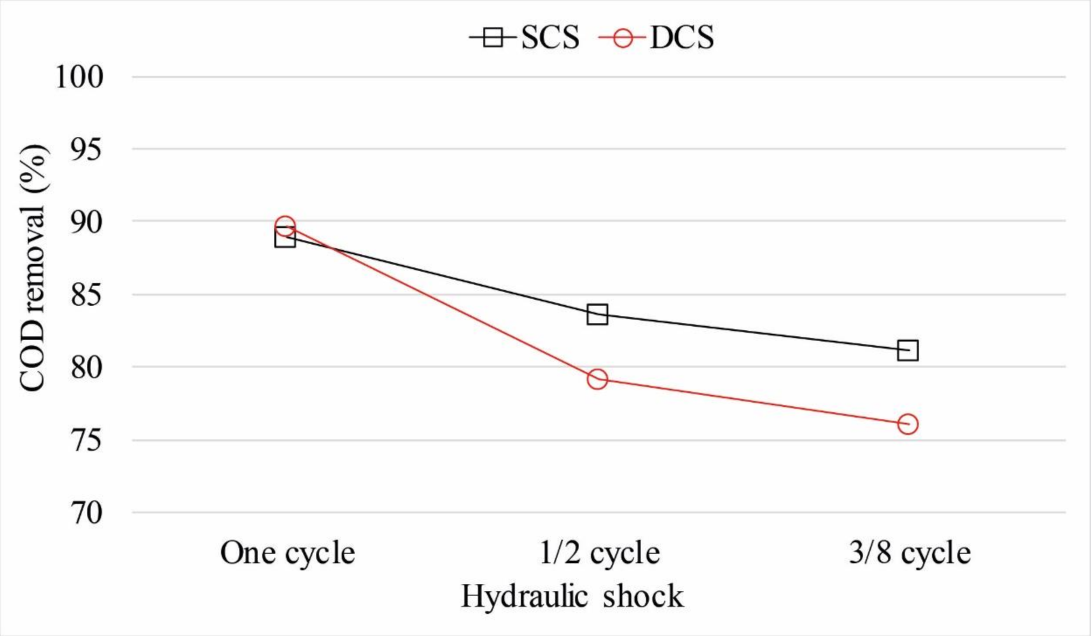



Figure 5.40: The effect of hydraulic shock on COD removal

*MLSS 4 g/l; Fill 15 min*

#### 5.1.8.2 The Effect of hydraulic shock on $NH_3$-N removal

The removal efficiency of $NH_3$-N during the hydraulic shocks is shown in Figure 5.41.The removal efficiency of $NH_3$-N for SCS and DCS during the steady-state operation was 93.2% and 92.9% respectively. After the first shock (1/2 cycle), the removal efficiency of $NH_3$-N for SCS and DCS dropped to 79.2% and 76.5% respectively. When the second shock (3/8 cycle) was applied, the removal efficiency of $NH_3$-N for SCS and DCS declined to 72.1% and 70.6% respectively.

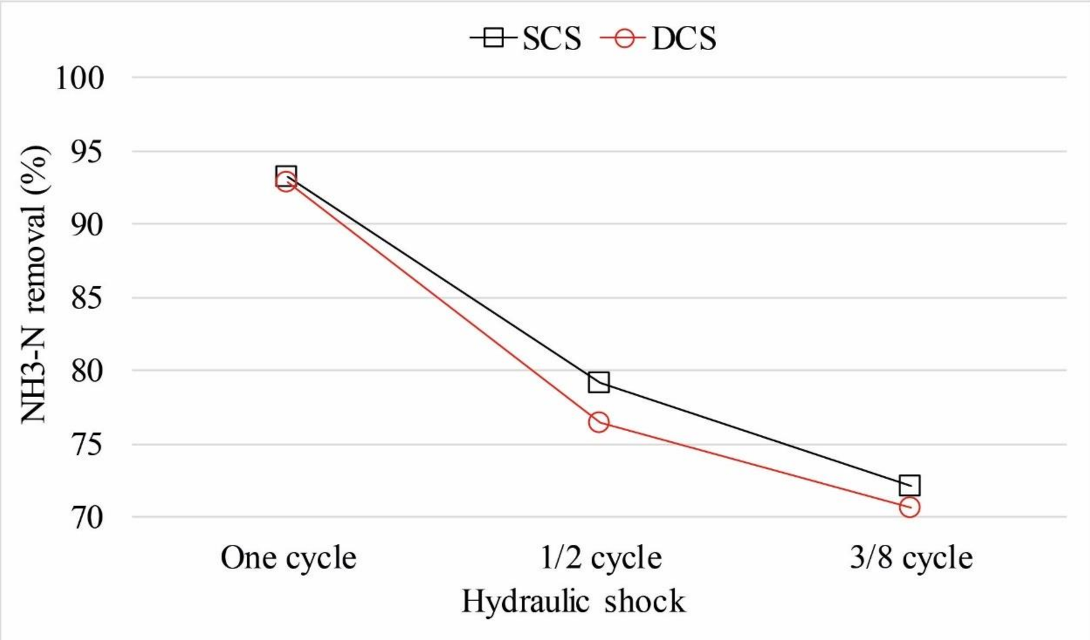



Figure 5.41: The effect of hydraulic shock on $NH_3$-N removal
*MLSS 4 g/l; Fill 15 min*

#### 5.1.8.3 **The Effect of hydraulic shock on $NO_3$-N removal**

Figure 5.42 shows the removal efficiency of $NO_3$-N during the hydraulic shocks. During the steady-state operation, the removal efficiency of $NO_3$-N for SCS and DCS was 96% and 97.1% respectively. After the first shock (1/2 cycle), the removal efficiency of $NO_3$-N for SCS and DCS dropped to 82.3% and 72.3% respectively. In addition, the removal efficiency of $NO_3$-N for SCS and DCS declined to 72.1% and 70.6% respectively, when the second shock (3/8 cycle) was applied.

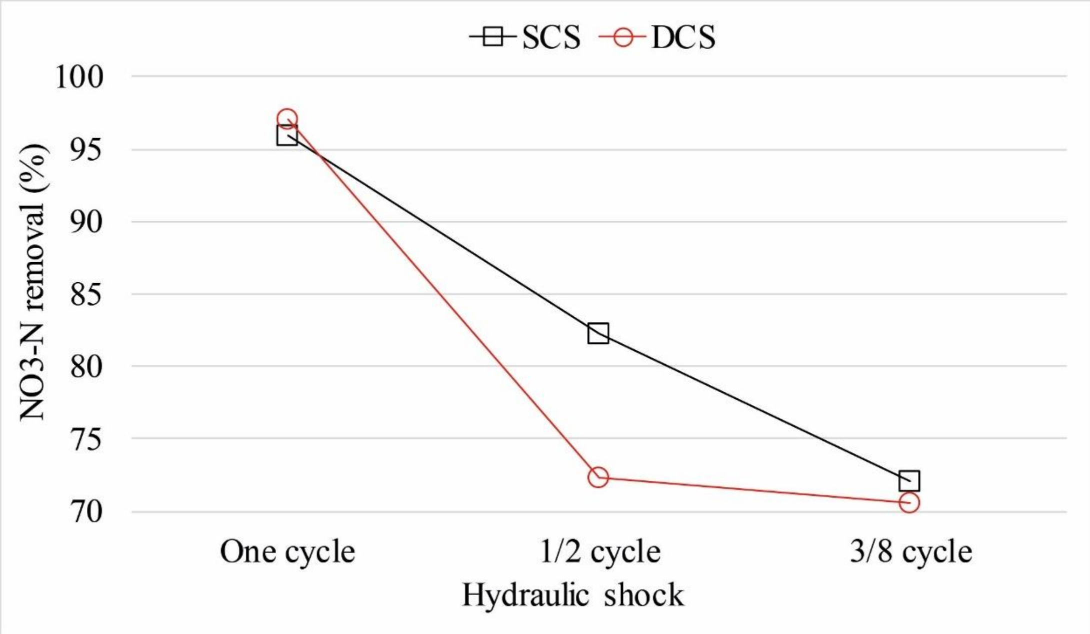



Figure 5.42: The effect of hydraulic shock on $NO_3$-N removal

*MLSS 4 g/l; Fill 15 min*

#### 5.1.8.4 **The Effect of hydraulic shock on $NO_2$-N removal**

Figure 5.43 shows the removal efficiency of $NO_2$-N during the hydraulic shocks. During the steady-state operation, the removal efficiency of $NO_2$-N for SCS and DCS was 95.2% and 95.1% respectively. After the first shock (1/2 cycle), the removal efficiency of $NO_2$-N for SCS and DCS dropped to 83.4% and 84.2% respectively. In addition, the removal efficiency of $NO_2$-N for SCS and DCS declined to 79.1% and 78.3% respectively, when the second shock (3/8 cycle) was applied.

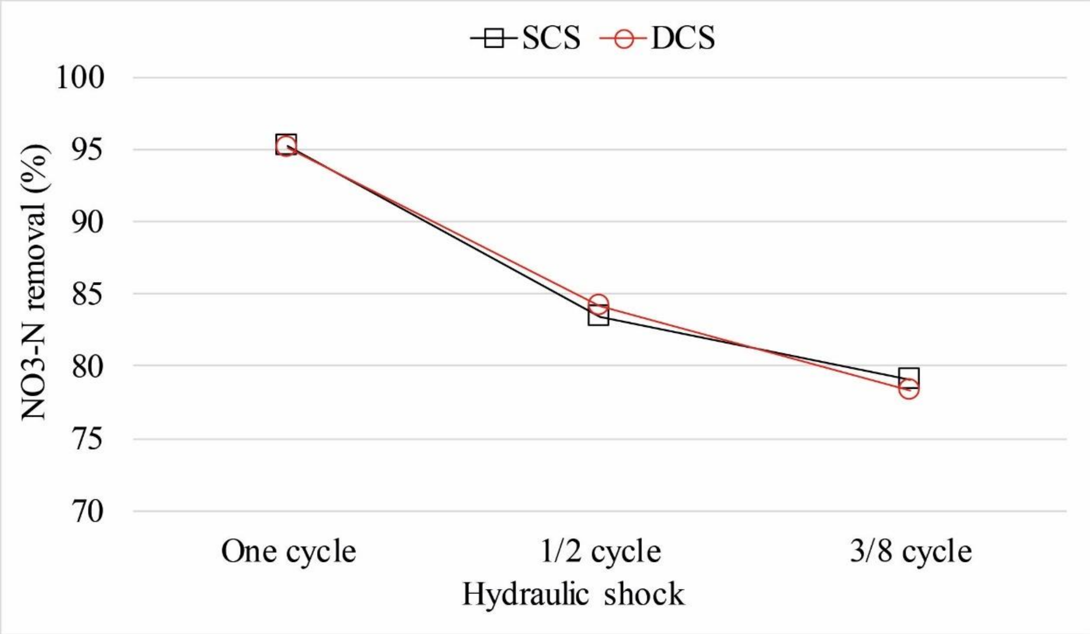



Figure 5.43: The effect of hydraulic shock on $NO_2$-N removal

*MLSS 4 g/l; Fill 15 min*

#### 5.1.8.5 **The Effect of hydraulic shock on sludge settleability**

SVI values during the hydraulic shocks are shown in Figure 5.44. During the steady-state operation, the SVI values for SCS and DCS were 35.2% and 34.6% respectively. After the first shock (1/2 cycle), the SVI values for SCS and DCS were raised to 39.1% and 41.7% respectively. In addition, the SVI values for SCS and DCS were further increased to 41.9% and 43.1% respectively, when the second shock (3/8 cycle) was applied.

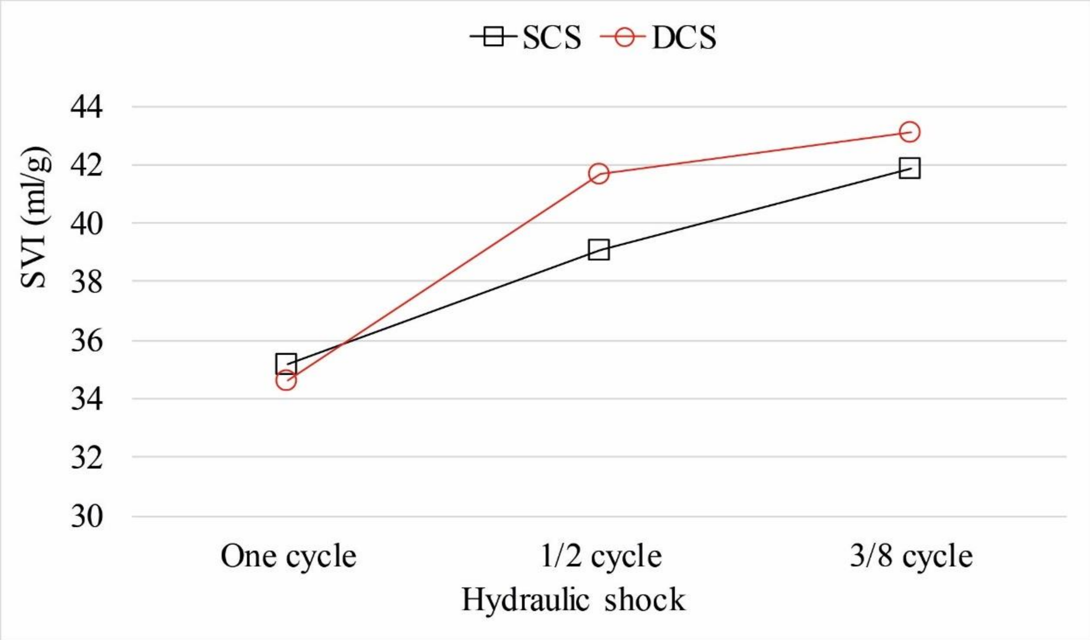



Figure 5.44: The effect of hydraulic shock on sludge settleability

*MLSS 4 g/l; Fill 15 min*

From the results above, it can be seen that the system could not handle a sudden hydraulic shock because the substrate degradation was not performed properly because of insufficient cycle time along with the high inhibition in the influent wastewater. This is in agreement with Mizzouri and Shaaban (2013), who treated petroleum refinery wastewater using a sequencing batch reactor, they reported that the removal efficiency of COD dropped from 90% to 78% in SCS. Also, it dropped from 90% to 72% in DCS. Nachaiyasit and Stuckey (1997) stated that small flocs could be washed out under low HRTs which could affect the treatment efficiency and settling performance of the SBR system.

## 5.2 Statistical analysis of the TSSBR operating condition optimisation

A multiple linear regression analysis was conducted to define whether the operating condition optimisation (OCO) of the TSSBR system statically affects the removal efficiency of the developed system. The value of $R^2$ for the model produced is 0.807, implying that the developed model was able to explain 80.7 % of the variation in the removal efficiency of the developed system according to operating condition optimisation values. The analysis of variance (Table 5-3) which tests whether or not the developed model is useful predictor to the removal efficacy of the TSSBR system gives a highly significant result (F = 82.99, sig = 0.000), indicating that the developed regression model provides accurate prediction to the system removal efficiency according to its inputs (OCO).

The removal efficiency was tested using seven OCO (15 minutes fill time, VER of 20%, Glucose OLR of 750 mg/l, Potassium-nitrate OLR of 50 mg/l, 6 hrs HRT, 4000 mg/l MLSS and unaerated fill mode). The results revealed that Potassium-nitrate OLR of 50 mg/l is associated with lower level of removal efficiency (Beta = -0.651, sig =0.000) comparing with MLSS concentration of 4000 mg/l. On the other hand, 15 minutes fill time is associated with higher level of removal efficiency (Beta = 0.328, sig = 0.000) comparing with MLSS concentration of 4000 mg/l. Besides, VER of 20%, Glucose OLR of 750 mg/l, 6 hrs HRT, 4000 mg/l MLSS and unaerated fill mode have a significant effect of the removal efficiency of the TSSBR as shown in Table 5-3.

Table 5-3: Multiple linear regression analysis for the TSSBR operating condition optimisation

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| R | 0.898 | | Std. Error | | | 1.867 | |
| $R^2$ | 0.807 | | Adjusted $R^2$ | | | 0.797 | |
| Analysis of variance | Sum of Squares | | df | Mean Square | | F | Sig. |
| Regression | 1736.921 | | 6 | 289.487 | | 82.99 | .000[b] |
| Residual | 415.050 | | 119 | 3.488 | | | |
| Total | 2151.972 | | 125 | | | | |
| Variables in equation | Unstandardized Coefficients | | Standardized Coefficients | t | Sig. | 95.0% Confidence Interval for B | |
| | B | Std. Error | Beta | | | Lower Bound | Upper Bound |
| (Constant) | 88.203 | .440 | | 200.374 | .000 | 87.331 | 89.074 |
| OCO=15 min | 3.879 | .623 | .328 | 6.231 | .000 | 2.646 | 5.111 |
| OCO=20% | 3.565 | .623 | .302 | 5.727 | .000 | 2.333 | 4.798 |
| OCO=50 mg/l | -7.691 | .623 | -.651 | -12.354 | .000 | -8.924 | -6.458 |
| OCO=6 hrs | 1.841 | .623 | .156 | 2.958 | .004 | .609 | 3.074 |
| OCO=750 mg/l | 2.587 | .623 | .219 | 4.156 | .000 | 1.355 | 3.820 |
| OCO=Unaerated | 2.378 | .623 | .201 | 3.819 | .000 | 1.145 | 3.610 |

Predictors: (Constant), OCO=15 min, OCO=20%, OCO=4000 mg/l, OCO=50 mg/l, OCO=6 hrs, OCO=750 mg/l.
Dependent Variable: Removal.

## 5.3 TSSBR performance under the optimal conditions

After obtaining the optimal operating conditions, the TSSBR system has operated under these conditions, which are shown in

Table 5-4 for three months, the treatment efficiency of COD and nitrogen compounds along with settling performance are shown below.

Table 5-4: TSSBR optimal operating conditions

| Operating condition | Unite | Value |
|---|---|---|
| MLSS | mg/l | 3000-4000 |
| HRT | hrs | 6 |
| Fill mode | - | Unaerated |
| Fill time | min | 15 |
| VER | % | 20 |
| OLR (glucose) | mg/l | 750-1000 |
| OLR (potassium-nitrate) | mg/l | 50-150 |

### 5.3.1 COD removal efficiency

The efficiency of the removal of COD for the TSSBR under the optimal operating conditions is shown in Figure 5.45. The minimum, maximum and the average values of COD removal efficiency of the TSSBR system over three months of operation were 82%, 96.5% and 95% respectively, with the average effluent of 32.4 mg/l.

When the operating conditions for the TSSBR optimised individually, the average removal efficiency of COD was between 90% and 94%. Then, after operating the TSSBR under the optimal values of all the operating conditions studied in this study, the average COD removal has risen to 95% for three months of operation, which means that operating the TSSBR under these condition has a significant effect on the removal efficiency of COD.

Many reasons lie behind this improvement, such as operating the system under the ultimate range of MLSS that contains a certain amount of active bacteria responsible for biodegrading the organic matter efficiently. By studying activated sludge process combined with biofilm cultivation (Wanner et al., 1998) agreed with the previous statement, he reported that the removal efficiency of COD was proportionally related to the concentration of MLSS. In this research, 3000 mg/l to 4000 mg/l was the optimal MLSS range which led to removing more than 95% of the influent COD concentration.

HRT in another important operating parameter which could directly affect the removal efficiency of COD. By studying the effect of hydraulic retention time and filling time on simultaneous biodegradation of phenol, resorcinol and catechol in a sequencing batch reactor, Thakur et al. (2013b) stated that higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates. Abu Hasan et al. (2016), who studied the removal of ibuprofen, ketoprofen, COD and nitrogen compounds from pharmaceutical wastewater using aerobic suspension-sequencing batch reactor (ASSBR), achieved up to 89% removal efficiency for COD at the end of 24 h HRT. In this research, 6 h HRT was the optimal HRT value, which led to removing more than 95% of the influent COD concentration.

In addition, fill modes and fill time are two important parameters that might increase the efficiency of the system. Although the results of this study showed that there is no effect of feeding modes on the treatment efficiency of COD, the TSSBR was operated under un-aerated fill mode because it showed better settling performance. By evaluating an SBR performance with aerated and un-aerated fill periods in treating phenol-containing wastewater, Chan and Lim (2007), stated that both periods (aerated and un-aerated fill) were capable of achieving consistently an effluent quality of less than 100 mg/L COD concentrations. In this research, 15 minutes was the optimal feeding time range, which led to removing more than 95% of the influent COD concentration. Damasceno et al. (2007) reported that longer feeding time is better for biodegrading a high concentration of COD. However, Thakur et al. (2013b) achieved negative results when increasing the feeding time. While Sahinkaya and Dilek (2007) found no effect of feeding time on the nutrient removal efficiency.

Moreover, VER is another important parameter which could affect the treatment efficiency of the system. In this research, 20% VER was the optimal range, which led to removing more than 95% of the influent COD concentration. There are many researchers who studied the effect of VER on the removal efficiency such as Arnz et al. (2000), who studied simultaneous loading and draining as a means to enhance the efficacy of sequencing biofilm batch reactors (SBBR), they found out that at a volumetric exchange rate of 68% in the lab-scale SBBR achieved high COD removal rates. Zielinska et al. (2012) also reached up to 93% of COD removal with 50% volumetric exchange rate when studying nitrogen removal from wastewater and bacterial diversity in the activated sludge at different COD/N ratios and dissolved oxygen concentrations. In addition, Tsang et al. (2007), stated that high VER results in poor effluent quality.

Finally, OLR is another important parameter which could directly affect the removal efficiency of the system. In this research, 750 to 1000 mg/l glucose loading rate; and 50 to 150 mg/l potassium nitrate loading rate were the optimal OLR range, which led to removing more than 95% of the influent COD concentration. Liu and Tay (2004) stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

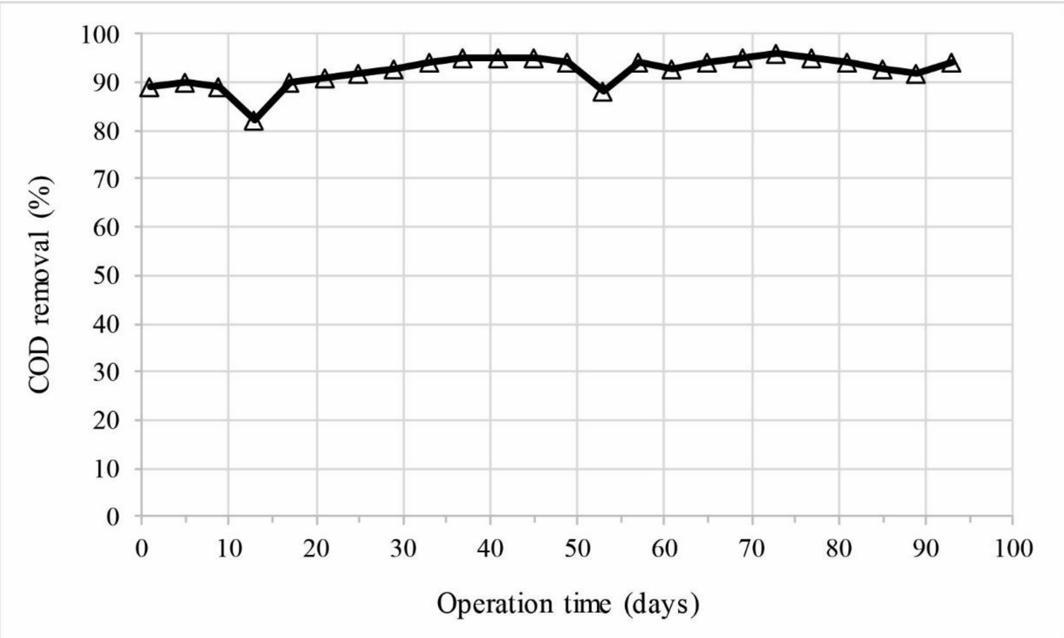



Figure 5.45: COD removal efficiency of TSSBR under the optimal conditions

### 5.3.2 $NH_3$-N removal efficiency

The efficiency of the removal of $NH_3$-N for the TSSBR under the optimal operating conditions is shown in Figure 5.46. The minimum, maximum and the average values of $NH_3$-N removal efficiency of the TSSBR system over three months of operation were 82.5%, 93% and 90.9% respectively, with the average effluent of 0.79 mg/l.

When the operating conditions for the TSSBR were optimised individually, the average removal efficiency of $NH_3$-N was between 90% and 96%. Then, after operating the TSSBR under the optimal values of all the operating conditions studied in this study, the average $NH_3$-N removal was 90.9% for three months of operation, which means that operating the TSSBR under these conditions has a negative effect on the removal efficiency of $NH_3$-N and this might be due to some interference between these combined conditions.

The MLSS parameter plays a critical role in treatment performance of the system. In this research, 3000 mg/l to 4000 mg/l were the optimal MLSS range, which led to removing more than 90% of the influent $NH_3$-N concentration. However, sometimes the MLSS goes above the 4000 mg/l due to the OLR applied, this leads to a reduction in removal efficiency when increasing MLSS. This is because of an increase in the microorganisms in the system leading to a decrease in the food/microorganisms (F/M) ratio, reducing microorganism activity.

HRT is another important operating parameter which could directly affect the removal efficiency of $NH_3$-N. In this research, 6 h HRT was the optimal HRT value, which led to removing more than 90% of the influent $NH_3$-N concentration. (Abu Hasan et al.,

2016) achieved up to 89%, 96% and 92.5% removal efficiency for COD, $NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT.

In addition, fill modes and fill time are two important parameters that might increase the efficiency of the system. Although the results of this study showed that there is no effect of feeding modes on the treatment efficiency of COD, the TSSBR was operated under un-aerated fill mode because it showed better settling performance. Liu et al. (2013) who studied the effect of fill and aeration modes and influent COD/N ratios on the nitrogen removal performance, stated that un-aerated fill could have relatively higher $NH^{4+}$-N removal due to stronger microbial activity under the anaerobic conditions. In this research, 15 minutes was the optimal feeding time range, which led to removing more than 90% of the influent $NH_3$-N concentration.

Moreover, VER is another important parameter, which could affect the treatment efficiency of the system. In this research, 20% VER was the optimal range which led to removing more than 90% of the influent $NH_3$-N concentration. Bernat et al. (2011) studied the removal of nitrogen from wastewater with a low COD/N ratio at a low oxygen concentration, during the experiment; three series differing in the volumetric exchange rate (10%, 30% and 50%) were conducted. At a volumetric exchange rate of 10% and 30%, total ammonia was removed in the first aeration phase. At the highest volumetric exchange rate of 50%, a significant increase in the ammonia nitrogen concentration at the beginning of the SBR cycle to about 90 mg $NH_3$-N/L resulted in only about 80% of ammonia nitrogen being oxidized in the first aeration phase. Complete oxidation occurred in the second aeration phase. In addition, Zielinska et al. (2012) stated that with 50% volumetric exchange rate, the ammonia concentration in the effluent did not exceed 0.5 mg/l. While, Tsang et al. (2007), stated that high VER results in poor effluent quality.

Finally, OLR is another important parameter which could directly affect the removal efficiency of the system. In this research, 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate were the optimal OLR range, which led to removing more than 90% of the influent $NH_3$-N concentration. Liu and Tay (2004) stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

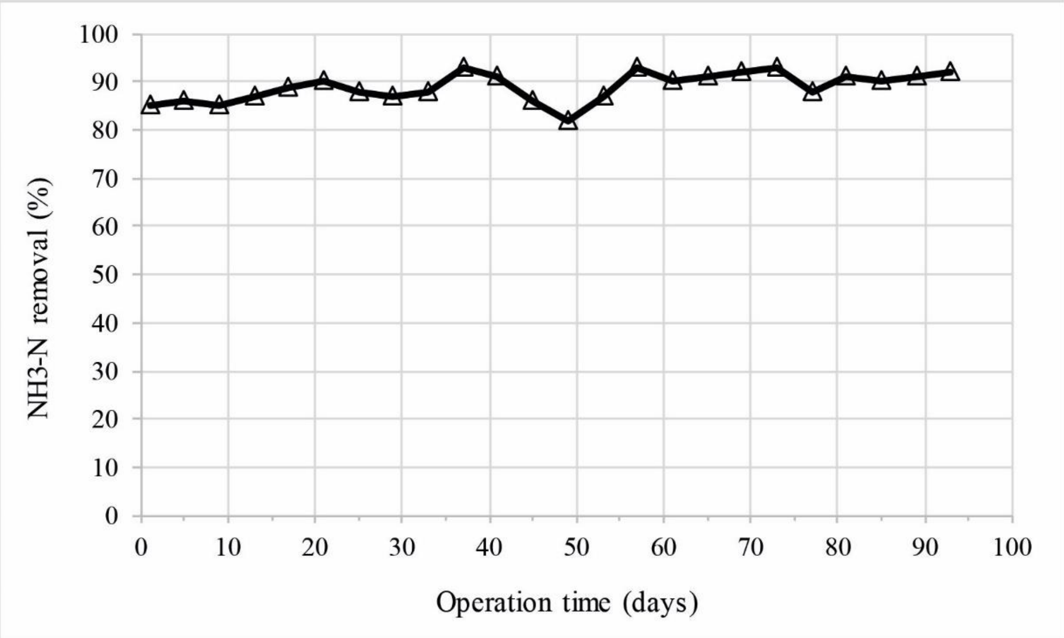



Figure 5.46: NH3-N removal efficiency of TSSBR under the optimal conditions

### 5.3.3 **$NO_3$-N removal efficiency**

The efficiency of the removal of $NO_3$-N for the TSSBR under the optimal operating conditions is shown in Figure 5.47. The minimum, maximum and the average values of $NO_3$-N removal efficiency of the TSSBR system over three months of operation were 88%, 98% and 96.1% respectively, with the average effluent of 0.75 mg/l.

When the operating conditions for the TSSBR were optimised individually, the average removal efficiency of $NO_3$-N was between 89.1% and 95.6%. Then, after operating the TSSBR under the optimal values of all the operating conditions studied in this study, the average $NO_3$-N removal has risen to 96.1% for three months of operation, which means that operating the TSSBR under these condition has a significant effect on the removal efficiency of $NO_3$-N.

A few reasons might be behind this improvement, such as operating the system under the ultimate range of MLSS that has a direct effect on the treatment efficiency of the system. In this research, 3000 mg/l to 4000 mg/l was the optimal MLSS range, which led to removing more than 96% of the influent $NO_3$-N concentration. Maintaining the MLSS within this range, led to an increase in removal efficiency due to keeping the microorganism active by offering the microorganisms in the system the optimal food/microorganisms (F/M) ratio.

HRT is another important operating parameter which could directly affect the removal efficiency of $NO_3$-N. In this research, 6 h HRT was the optimal HRT value, which led to removing more than 96% of the influent $NO_3$-N concentration. Thakur et al. (2013b) stated that higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates. In addition, Abu Hasan et al. (2016), achieved up to 89%, 96% and 92.5% removal efficiency for COD, $NH_3$–N and $NO_3$–N respectively at the end of 24 h HRT.

In addition, fill modes and fill time are two important parameters that might increase the efficiency of the system. Although the results of this study showed that there is no effect of feeding modes on the treatment efficiency of $NO_3$-N, the TSSBR was operated under un-aerated fill mode because it showed better settling performance. In this research, 15 minutes was the optimal feeding time range, which led to removing more than 96% of the influent $NO_3$-N concentration. Liu et al. (2013) stated that aerated fill could strengthen the nitrogen removal with the presence of carbon source, but no statistically significant effect of intermittent aerated fill on nitrogen removal was observed with the COD/N ratio of 2.5. In addition, Yu et al. (1996) who studied the effect of fill mode on the performance of sequencing-batch reactors treating various wastewaters, stated that in the aerated fill, the $NO_3$-N concentration was 68 mg/l, while in the unaerated fill, the $NO_3$-N concentration was only 45 mg/l, based on this, it is clear that there had been an effective denitrification during the un-aerated fill mode probably due to the different availability of organic carbon sources to denitrifiers during the second anoxic mixing period.

Moreover, VER is another important parameter which could affect the treatment efficiency of the system. In this research, 20% VER was the optimal range, which led to removing more than 96% of the influent $NO_3$-N concentration. Tsang et al. (2007), stated that high VER results in poor effluent quality. In addition, Zielinska et al. (2012) stated that with 50% volumetric exchange rate, the effluent nitrate concentration was 33.2 mg/l.

Finally, OLR is another important parameter which could directly affect the removal efficiency of the system. In this research, 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate were the optimal OLR range, which led to removing more than 96% of the influent $NO_3$-N concentration. Liu and Tay (2004) stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

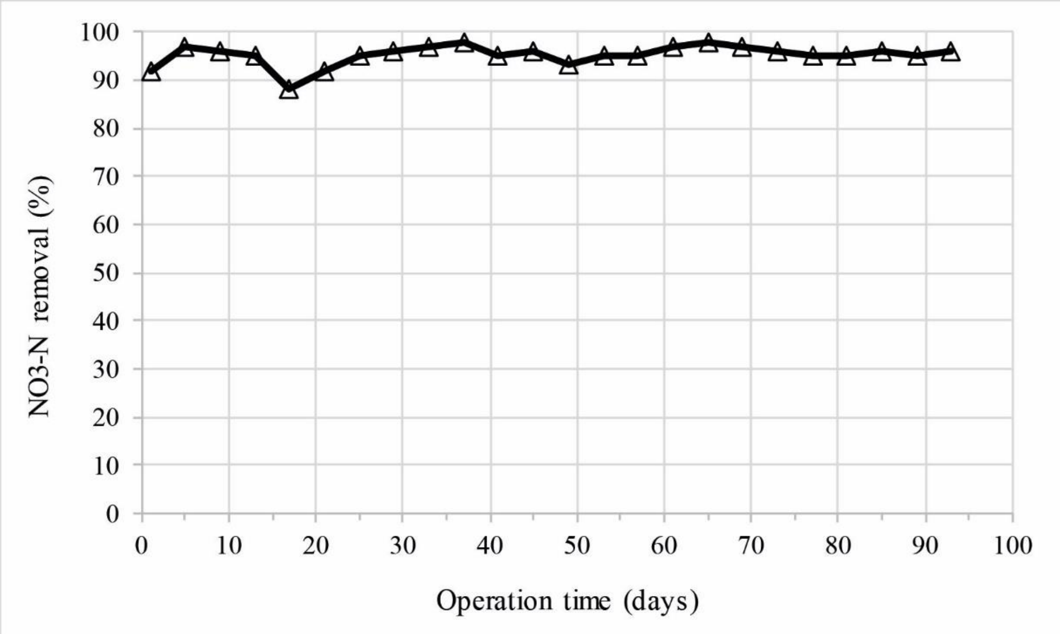



Figure 5.47: NO3-N removal efficiency of TSSBR under the optimal conditions

### 5.3.4 $NO_2$-N removal efficiency

The efficiency of the removal of $NO_2$-N for the TSSBR under the optimal operating conditions is shown in Figure 5.48. The minimum, maximum and the average values of $NO_2$-N removal efficiency of the TSSBR system over three months of operation were 82.1%, 99% and 93.2% respectively, with the average effluent of 0.71 mg/l.

When the operating conditions for the TSSBR were optimised individually, the average removal efficiency of $NO_2$-N was between 91.1% and 97.7%. Then, after operating the TSSBR under the optimal values of all the operating conditions studied in this study, the average $NO_2$-N removal was to 90.9% for three months of operation, which means that operating the TSSBR under these conditions has a negative effect on the removal efficiency of $NO_2$-N and this might be due to some interference between these combined conditions.

Many reasons lie behind these results, such as operating the system under the ultimate range of MLSS that contains a certain amount of active bacteria responsible for biodegrading the organic matter effectively. In this research, 3000 mg/l to 4000 mg/l was the optimal MLSS range, which led to removing more than 90% of the influent $NO_2$-N concentration. However, sometimes the MLSS goes above the 4000 mg/l due to the OLR applied, this leads to a reduction in removal efficiency when increasing MLSS. This is because of an increase in the microorganisms in the system leading to a decrease in the food/microorganisms (F/M) ratio, reducing microorganism activity.

HRT is another important operating parameter that could directly affect the removal efficiency of $NO_2$-N. In this research, 6 h HRT was the optimal HRT value that led to removing more than 90% of the influent $NO_2$-N concentration.

In addition, fill modes and fill time are two important parameters that might increase the efficiency of the system. Although the results of this study showed that there is no effect of feeding modes on the treatment efficiency of $NO_2$-N, the TSSBR was operated under un-aerated fill mode because it showed better settling performance. Yu et al. (1996) who studied the effect of fill mode on the performance of sequencing-batch reactors treating various wastewaters, stated that both aerated and un-aerated fill were significant in terms of nitrogen compounds removal rates.

Moreover, VER is another important parameter which could affect the treatment efficiency of the system. In this research, 20% VER was the optimal range, which led to removing more than 95% of the influent $NO_2$-N concentration.

Finally, OLR is another important parameter which could directly affect the removal efficiency of the system. In this research, 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate were the optimal OLR range, which led to removing more than 95% of the influent $NO_2$-N concentration. Liu and Tay (2004) stated that at high ORL, the removal of COD and nitrogen would be decreased. However, Sato et al. (2016) reached high COD and nitrogen compounds removal rates even under high ORL.

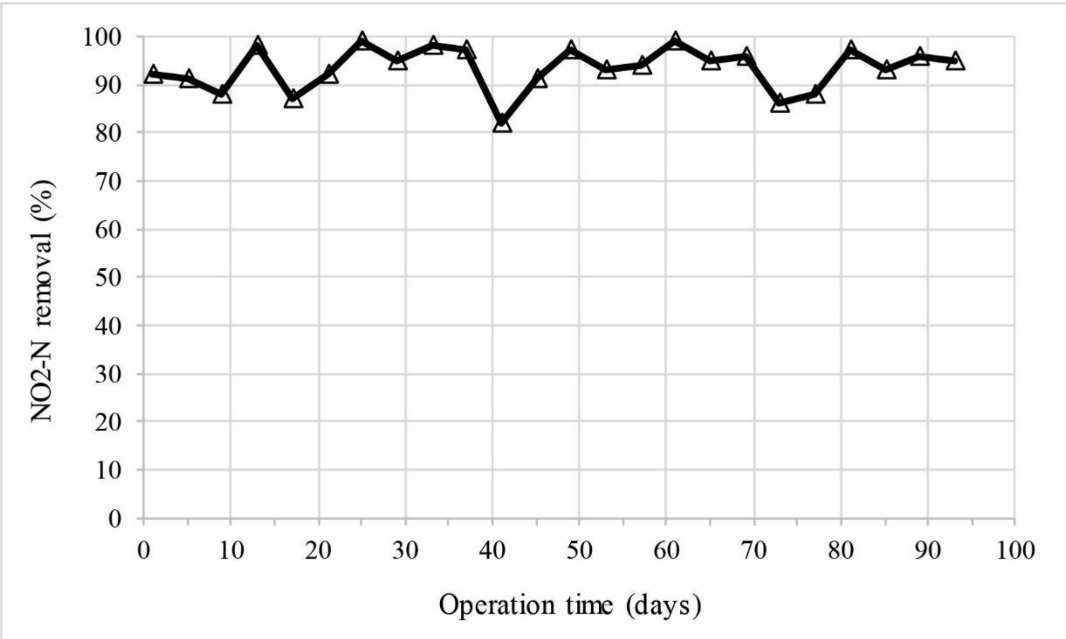



Figure 5.48: NO2-N removal efficiency of TSSBR under the optimal conditions

### 5.3.5 Sludge settleability

The SVI values for the TSSBR under the optimal operating conditions are shown in Figure 5.49. The minimum, maximum and the average values of SVI of the TSSBR system over three months of operation were 27.5 ml/g, 35 ml/g and 30.9 ml/g respectively.

When the operating conditions for the TSSBR were optimised individually, the average SVI value was between 31.9 ml/g and 45 ml/g. Then, after operating the TSSBR under the optimal values of all the operating conditions studied in this study, the average SVI value has risen to 30.9 ml/g for three months of operation, which means that operating the TSSBR under these condition has a significant effect on the solids' settleability.

Many reasons behind this improvement, such as operating the system under the ultimate range of MLSS that can directly affect the settling performance. In this research, 3000 mg/l to 4000 mg/l were the optimal MLSS range, which led to improving the settling performance, SVI measured 30.9 under this range of MLSS. Tsang et al. (2007) stated that a smaller amount of biomass could settle better in the bioreactor system than a large amount.

HRT is another important operating parameter, which could directly affect the settling performance. In this research, 6 h HRT was the optimal HRT value that led to improving the settling performance, SVI measured 30.9 under this value of HRT. Below this value of HRT could affect negatively on the settling performance, Cervantes (2009), stated that reducing the HRT will increase the biomass concentration, and thus sludge will take longer to settle.

In addition, fill modes and fill time are two important parameters that might increase the efficiency of the system. Although the results of this study showed that there is no effect of feeding modes on the treatment efficiency of COD and nitrogen compounds, the TSSBR was operated under un-aerated fill mode because it showed better settling performance. The improvement in the settling performance in the un-aerated fill over the aerated fill could be because the aeration enhances the growth rate of several kinds of bacteria which would increase the biomass in the reactor and slow down the solids' settling performance (Rodriguez-Perez and Fermoso, 2016). In addition, while studying the effect of fill mode on the performance of sequencing-batch reactors treating various wastewaters, Yu et al. (1996) stated that at low influent phenol concentrations, the SBR with an un-aerated fill was better than the SBR with an aerated fill in terms of sludge settleability, as aeration encouraged the growth of filamentous bacteria under low substrate concentration conditions. Chan and Lim (2007) evaluated an SBR performance with aerated and un-aerated fill periods in treating phenol-containing wastewater, the results obtained showed that the mean SVI values were 93 and 89 mL/g for reactors aerated and un-aerated fill respectively, indicating good sludge settleability when the influent phenol concentration was at 100 mg/L, the good sludge settleability in the un-aerated fill reactor could be explained by the anaerobic conditions prevailing in the reactor which favoured floc forming organisms.

Moreover, VER is another important parameter which could affect the treatment efficiency of the system. In this research, 20% VER was the optimal range, which led to improving the settling performance.

Finally, OLR is another important parameter which could directly affect the removal efficiency of the system. In this research, 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate were the optimal OLR range, which led to improving the settling performance. Xu et al. (2014) stated that increasing the OLR will lead to a proportional increase in biomass concentration, which will result in high SVI and the settleability of the solids will decrease. This agreed with Bassin et al. (2016), who reported an increase in the concentration of suspended solids when the initial concentration of COD was increased, which led to an increase in the SVI and a subsequent drop in the solids' settleability.

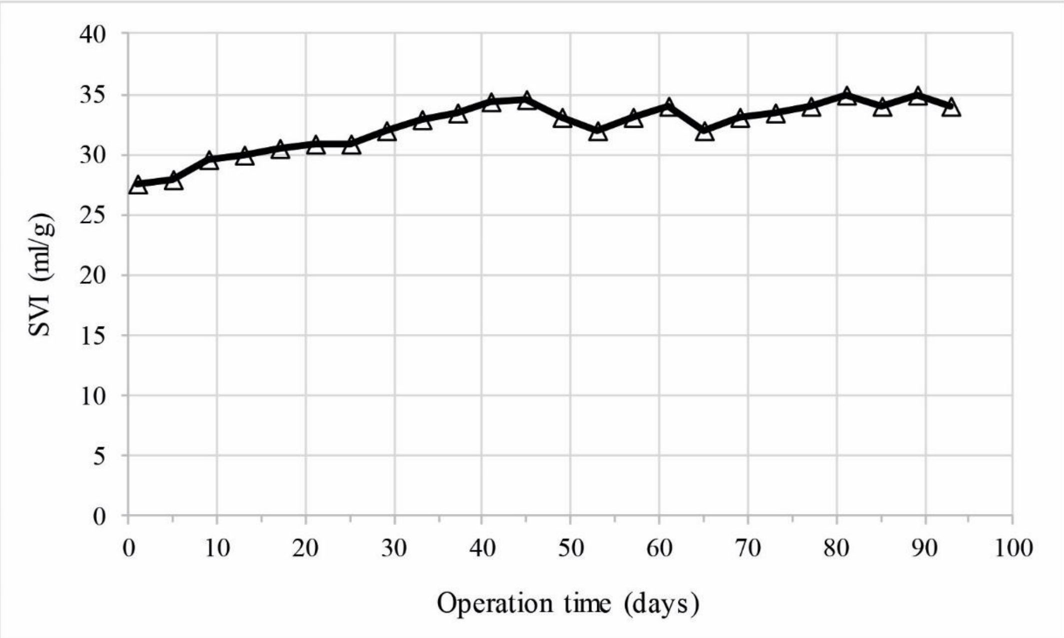



Figure 5.49: SVI values of TSSBR under the optimal conditions

## 5.4 Chapter summary

In the first part of this chapter, seven SBR operating parameters (mixed liquor suspended solids, hydraulic retention time, fill conditions, fill time, volumetric exchange rate, organic loading rate and hydraulic shock) have been studied, and their effects on the removal efficiency and settling performance were examined and the optimal ranges of each parameter were obtained.

The optimal MLSS range was 3000 mg/l to 4000 mg/l. The optimal HRT value was 6 h. Un-aerated fill was better than the aerated fill, and 15 minutes was the optimal feeding time. The optimal VER value was 20%. The optimal OLR ranges were 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate.

In the second part of this chapter, the TSSBR was operated under the obtained optimal operating conditions to find the best performance of the TSSBR. After operating the TSSBR under the optimal operating conditions, the treatment efficiency of COD and $NO_3$-N have been improved dramatically. Although the removal efficiency of $NH_3$-N and $NO_2$-N did not improve, the removal efficiency of both is more than 90%, which is considered a good treatment efficiency for the system. In addition, the settling performance of the TSSBR was significantly improved after operating the system under the optimal operating conditions. In the next chapter (Chapter 6), the conclusions and recommendations for the future work will be mentioned.

# CHAPTER 6

# Conclusions and recommendations

## 6.1 Conclusions

The removal efficiency of COD and nitrogen compound, along with the settling performance of normal operating sequencing batch reactors, were determined and compared with the performance of a novel, two-stage, settling sequencing batch reactor, to improve the sludge settleability in the SBR as this is considered a major drawback for SBRs.

In addition, the operating conditions of TSSBR were optimised in terms of (mixed liquor suspended solids, hydraulic retention time, fill conditions, fill time, volumetric exchange rate, organic loading rate and hydraulic shock), and their effects on the removal efficiency and settling performance were examined and the optimal ranges of each parameter were found.

The results obtained from this research showed that a TSSBR with a 5.5 h cycle time improved sludge settleability and enhanced nitrogen compounds' removal efficiency, while COD removal efficiency for the NOSBR and TSSBR remained the same. The morphological characteristics of the sludge inside the TSSBR reactor, showed better and homogeneous growth of filamentous bacteria in comparison to that in the NOSBR which showed overgrowth of filamentous bacteria. Finally, a significant linear relationship between the total filament length and SVI was found, this having a direct effect on sludge settleability.

In addition, after operating the TSSBR under the optimal operating conditions, the treatment efficiency of COD and $NO_3$-N have been improved dramatically. Although the removal efficiency of $NH_3$-N and $NO_2$-N did not improve, the removal efficiency of both is more than 90%, which is considered a good treatment efficiency for the TSSBR system. In addition, the settling performance of the TSSBR was significantly improved after operating the system under the optimal operating conditions.

A summary of the main conclusions for the research work presented in this thesis are listed as follows:

1. The average efficiency for the removal of COD in the NOSBR and TSSBR was 93.7% and 93.1%, respectively, the average effluent was 54.83 mg/l and 34.7, respectively.

2. The average efficiency of removal of $NH_3$-N for the NOSBR was 76.6% with an average effluent of 1.87 mg/l. While, the average efficiency of the removal of $NH_3$-N for the TSSBR was 89.2% with an average effluent of 0.85 mg/l.

3. The average efficiency of removal of $NO_3$-N for the NOSBR was 86.4% with an average effluent of 2.41 mg/l. While the average efficiency of the removal of $NO_3$-N for the TSSBR was 95.2% with an average effluent of 0.81mg/l.

4. The average efficiency of removal of $NO_2$-N for the NOSBR was 87.3% with an average effluent of 2.23 mg/l. While the average efficiency of the removal of $NO_2$-N for the TSSBR was 96% with an average effluent of 0.75 mg/l.

5. The average SVI for TSSBR and NOSBR was 31.17 ml/g and 42.04 ml/g, respectively.

6. The average TL/MLSS for TSSBR and NOSBR was 1475.33 mm/mg and 1594.34 mm/mg, respectively while the average TL/Vol was 139.70 mm/µl and 221.79 mm/µl, respectively.

7. During the second and third months of operation, the settling ability of the NOSBR dropped due to the filamentous growth inside the reactor, while the morphological characteristics of the sludge inside the TSSBR reactor, have better and more homogenous growth of filamentous bacteria.

8. The optimal MLSS range was 3000 mg/l to 4000 mg/l, which led to removing more than 95%, 90%, 96% and 90% of the initial concentration of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N respectively. In this range of MLSS, the settling performance of TSSBR was significantly improved.

9. The optimal HRT value was 6 h, which led to removing more than 95%, 90%, 96% and 90% of the initial concentration of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N respectively. In this value of HRT, the settling performance of TSSBR was significantly improved.

10. Un-aerated fill was better than the aerated fill; the fill mode had no effect on the removal efficiency of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N. However, during the un-aerated fill, the settling performance of TSSBR was significantly improved.

11. The optimal feeding time was 15 minutes, which led to removing more than 95%, 90%, 96% and 90% of the initial concentration of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N respectively. In this time of feeding, the settling performance of TSSBR was significantly improved.

12. The optimal VER was 20%, which led to removing more than 95%, 90%, 96% and 90% of the initial concentration of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N respectively. In this VER value, the settling performance of TSSBR was significantly improved.

13. The optimal OLR ranges were 750 to 1000 mg/l glucose loading rate and 50 to 150 mg/l potassium nitrate loading rate. Within these ranges of OLR the settling performance of TSSBR was significantly improved, and more than 95%, 90%, 96% and 90% of the initial concentration of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N respectively was removed.

## 6.2 Recommendations for further works

Based on the experience gained during the course of this research, a number of possible future studies can be recommended, as listed below:

1. Bacterial identification of the mixed culture inside the TSSBR reactor to fully understand the bacterial culture in the TSSBR system that could help to improve the settling performance further.

2. Study different temperature ranges rather than the ambient temperature, as the temperature has an essential impact on the bacterial growth and consequently on settling performance.

3. Investigate the performance of TSSBR on real wastewater samples to see the system performance during wide range and different loads of pollutants.

4. Investigate the possibility of operating the TSSBR system depending on the online monitoring parameters (pH, DO and ORP). Instead of analysing the

parameters of COD, $NH_3$-N, $NO_3$-N and $NO_2$-N offsite, which is costly and time-consuming, a control system using online monitoring of the pH, DO and ORP could accurately detect the removal time for these parameters and could estimate the end of the treatment cycle.

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# Appendices

## Appendix I: MATLAB code for the morphological study

```
clear

clc

mkdir('output_figures');

 raw = dir(['10\*.jpg']);

 for i = 1:length(raw)

    a = imread([raw(i).folder '\' raw(i).name]);

    Igray = rgb2gray(a);

% This is to convert the original picture to grayscale

    level1 = 0.5;

    level2 = 0.6;

    Ithresh1 = im2bw(Igray,level1);

% This is for aggregate segmentation after converting the % picture from grayscale

% to binary

    Ithresh2 = im2bw(Igray,level2);

% This is for filamentous segmentation and debris alimentation after converting the

% picture from grayscale to binary

    h = figure(1);

    subplot(2,2,1),imshow(a);

    title('Orginal image');

    subplot(2,2,2),imshow(Igray);

    title('Grayscale image');

    subplot(2,2,3),imshow(Ithresh1);
```

```
  title('Binary 0.5');
  subplot(2,2,4),imshow(Ithresh2);
  % This is for plotting four pictures in one figure
  title('Binary 0.6');
  % This is for naming the output file so that it will be consistent with the
  % original name
  name_length = length(raw(i).name) - 4;
     outfile = ['output_figures\' raw(i).name(1:name_length)];
  print ('-dtiff','-r500' ,outfile)
  close(h)
end
```

**Appendix II: List of Journal Publications**

**Published journal papers:**

1. Ali W. Alattabi, Clare B. Harris, Rafid M. Alkhaddar, Khalid Hashim, Montserrat Ortoneda-Pedrola, David Phipps, Improving sludge settleability by introducing an innovative two-stage settling sequencing batch reactor, Journal of water process engineering, 20 (2017) 207-216.

2. Ali W. Alattabi, Clare Harris, Rafid Alkhaddar, Montserrat Ortoneda-Pedrola, Ali Alzeyadi, An investigation into the effects of MLSS on the effluent quality and sludge settleability in an aerobic-anoxic sequencing batch reactor (AASBR), Journal of Water Process Engineering (2017), http://dx.doi.org/10.1016/j.jwpe.2017.08.017.

3. Ali Alattabi , Clare Harris, Rafid Alkhaddar, Ali Alzeyadi and Khalid Hashim, Treatment of Residential Complexes' Wastewater using Environmentally Friendly Technology, Procedia engineering, 196 (2017) 792–799.

4. Ali Alattabi , Clare Harris, Rafid Alkhaddar, Ali Alzeyadi and Muhammad Abdulredha, Online Monitoring of a Sequencing Batch Reactor Treating Domestic Wastewater, Procedia Engineering 196 (2017) 800–807.

5. Alattabi A. W., Hashim, K., Jafer, H. & Alzeyadi, A., Removal of Nitrogen Compounds from Industrial Wastewater Using Sequencing Batch Reactor: The Effects of React Time. International Journal of Civil, Environmental, Structural, Construction and Architectural Engineering, Vol. 10(7), 964-967, 2016

## Appendix III: List of Conference Publications

### Presented conference papers:

1. Alattabi, A. W., Harris, C. B. & Alkhaddar, R. M., The effect of sludge wasting on the treatment efficiency of petroleum refinery wastewater (PRW) in an aerobic suspension-sequencing batch reactor (ASSBR), LJMU, Faculty research week, 2015, Liverpool, UK.

2. Alattabi, A. W., Harris, C. B. & Alkhaddar, R. M., The effects of different mixed liquor suspended solids concentrations on the sludge characteristics in a SBR, LJMU, Faculty research week, 235-240, 2016, Liverpool, UK.

3. Alattabi, A. W., Harris, C. B., Alkhaddar, R. M. & Alzeyadi, A, The relationship between operating condition and sludge wasting of an aerobic suspension sequencing batch reactor (ASSBR) treating phenolic wastewater. The Second BUiD Doctoral Research Conference, 417-423, 2016, Dubai, UAE.

4. Alattabi, A. W. N., Harris, C. B., Alkhaddar, R. M. & Alzeyadi, A., The effects of different MLSS concentrations on the sludge characteristics and effluent quality in an ASSBR under low temperature, 1st International Conference on Sustainable Water Processing, Spain, 2016.

5. Alattabi, A. W. N., Harris, C. B., Alkhaddar, R. M. & Alzeyadi, A., The effects of hydraulic retention time on the sludge characteristics and effluent quality in an ASSBR, International Conference for Students on Applied Engineering (ICSAE), 458-462, 2016. doi: 10.1109/ICSAE.2016.7810235, Newcastle, UK.

6. Ali Alattabi, Clare Harris, Rafid Alkhaddar and Ali Alzeyadi, The effects of potassium nitrate loading rate on the sludge settling and effluent quality in an ASSBR, Cutting Edge Postgraduate Research Conference 2017, 2017, Edge Hill, UK.

7. Ali Alattabi, Clare Harris, Rafid Alkhaddar and Ali Alzeyadi, The impact of organic loading rate on the removal efficiency of nitrate-nitrogen using

sequencing batch reactor,  LJMU, Faculty research week, 2017, Liverpool, UK.

8. Ali Alattabi, Clare Harris, Rafid Alkhaddar and Ali Alzeyadi, Ammonia removal using sequencing batch reactor: the effects of organic loading rate, The third BUiD doctoral research conference, 2017, Dubai, UAE.

9. Ali W. Alattabi, Clare B. Harris, Rafid M. Alkhaddar, Khalid S. Hashim and Muhammad Abdulredha, Removal of nitrogen compounds under different loading of potassium nitrate in a SBR, The 2nd International Conference for Science & Arts, Hilla, Iraq, 2018.

10. Ali Alattabi, Clare Harris and Rafid Alkhaddar, Industrial wastewater treatment using two-stage settling sequencing batch reactor: the effects of feeding time, LJMU, Faculty research week, 2018, Liverpool, UK.

## Appendix IV: Awards and recognitions

- "Best Paper Award" at Faculty research week 2017, Liverpool John Moores University, UK, on the paper titled "The impact of organic loading rate on the removal efficiency of nitrate-nitrogen using sequencing batch reactor", May 2017.

- Medal of excellence from Iraqi minister of Higher education, for publishing more than 10 papers, January 2018.


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والبحث العلمي

LIVERPOOL
JOHN MOORES
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Faculty of Engineering & Technology

*The Award for Best Paper – winner - at Faculty Research Week 2017 is awarded to:*

Ali Al-Attabi

*Signed: Executive Dean*

26th MAY 2017

*Dated*