Novel Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS) to enhance nutrients removal from wastewater

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Abstract

Novel Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS) to enhance nutrients removal from wastewater

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This study aimed to improve nitrogen removal through developing an Electro-Integrated Fixed-film Activated Sludge reactor. Electrical current controlled redox for both aerobic and anoxic conditions in a single vessel. This complete-mixed reactor ensures the removal of nitrogen and phosphorous. The first phase of this research, which had two phases, focused on ammonia removal from the Integrated Fixed-film Activated Sludge reactor (IFAS) under aerobic conditions with an average dissolved oxygen level of 7 mg/L. Whereas, the second phase was focused on nutrient removal in a novel Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS). The results of this study showed a promising nitrification process in IFAS with high-efficiency ammonia-nitrogen removal (up to 96% under aerobic conditions) after ten months, although a significant increase in nitrate concentration was recorded. Similarly, the finding from the second phase of this study showed a 75% removal of ammonia-nitrogen with a nitrate-nitrogen concentration of 2.7 mg/L. Furthermore, EIFAS showed a successful removal of orthophosphate and chemical oxygen demand (COD), by 93% and 98% respectively.

This study used a novel EIFAS designed system for a successful wastewater treatment containing high concentrations of ammonia in a single tank. The technology follows sustainable development principles by decreasing footprint and energy consumption. It can be used in small and medium-sized wastewater treatment plants, upgrading existing facilities in order to prevent eutrophication of receptors.

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Dedications

I am truly grateful to my lovely parents for their emotional and financial support in every step of my life. Their unconditional love and encouragement have made me a better and more successful person.

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Glossary

Anammox	Anaerobic ammonium oxidation
AS	Activated sludge
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
DO	Dissolved oxygen
EC	Electro coagulation
EIFAS	Electro-Integrated Fixed-film Activated Sludge reactor
HRT	Hydraulic retention time
IFAS	Integrated Fixed-film Activated Sludge reactor
MBR	Membrane bioreactor
MBBR	Moving bed biofilm reactor
MLVSS	Mixed-liquor volatile suspended solids
NH_3^--N	Ammonia as nitrogen
NO_3^--N	Nitrate as nitrogen
ORP	Oxidation-reduction potential
PO_4^{3-}	Orthophosphate (Reactive phosphorus)
SA/AB	Sequential anoxic/aerobic bioreactor
SMEBR	Submerged membrane electro-bioreactor
SMP	Soluble microbial products
SRT	Sludge residence time
RBC	Rotating biological contactors
TKN	Total Kjeldahl nitrogen
TN	Total nitrogen
TSS	Total suspended solid

Chapter I

Introduction

1.1 Background

Water scarcity is a huge challenge that is threatening our existence. In dealing with this crisis, different methods like wastewater treatment and reuse have been adopted. The need for the treatment of wastewater is crucial, not just in meeting the increasing water demand but also in protecting the environment. Wastewater is rich in nutrients and treating it aims to ensure that water is safe to drink, and discharged effluent is harmless to the environment. Otherwise, several adverse impacts such as eutrophication and red tide could threaten our environment (Yang et al., 2010)

In Canada, 150 billion litres of sewage and untreated wastewater is annually discharged to water bodies (Government Canada, 2017). Consequently, effluent quality improvement has become necessary, not only in Quebec but also in other provinces. The 2018–2030 Québec Water Strategy ensures the public access to clean and potable water. This protection plan also guarantees management of water and aquatic environments such as rivers and lakes (Ministère du Développement durable, de l'Environnement et de la Lutte contre les, 2018). Undoubtedly, restoring and rebuilding aquatic systems is more costly than protecting them. Therefore, the allowable quality of discarded wastewater to surface and underground waters must be met. Considering that nutrient removal creates an improvement in wastewater quality, nutrient-removal-systems are inevitably important units in wastewater treatment plants.

Nutrient removal units are considered tertiary treatment processes in modern wastewater plant design. Biological treatment, adsorption, flocculation and coagulation, and electrokinetic systems are examples of treatment methods applied in tertiary treatment units (Zhang et al., 2014). Therefore, pros and cons of each method must be considered before plant design and construction.

Common wastewater treatment processes, e.g. membrane bioreactors (MBR), activated sludge systems (AS), and clarifier-based biological processes are able to partially remove nutrients; like ammonia and phosphorus. Nevertheless, these processes are limited by drawbacks, which in turn affect the overall performance. MBR is considered an established wastewater treatment process, which is immensely affected by membrane fouling (Drews et al., 2005). Activated sludge and clarifier-based processes require considerable space and chemical additives. Moreover, this process is costly to build and operate, requires high-energy consumption and sludge treatment (Massoud et al., 2009).

Conventional wastewater treatment facilities consist of nitrate and phosphorus removal units other than aerobic ammonia oxidation tanks. Some facilities have built additional compartments in the denitrification process in order to convert nitrates into gaseous nitrogen in anoxic conditions. Enhanced Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS) is a novel design in which aerobic and anoxic conditions occur in the same reactor. Consequently, complete nitrogen removal and effluent's acceptable quality is ensured.

1.2 Motivation

The majority of cities in Quebec use aerated ponds or lagoons in where nitrification takes place, followed by several anoxic and anaerobic ponds to remove by-products of the nitrification process. Moreover, coagulation reaction removes phosphorus in a settlement tank. This physical-chemical reaction requires chemical additives.

Due to population growth, competition rises over land between residential and municipal purposes. The enhancement of existing units is an effective alternative that reduces CAPEX of new units, saving energy and space, and responding to the demand of corresponding areas.

Integrated Fixed-film Activated Sludge (IFAS) has been applied in the past, and creates additional nutrient and suspended particle removal. It builds up the contact surface of microorganisms with nutrients and oxygen. Consequently, it improves system efficiency without required vast extra capital and operational expenses. In addition, inserting an electrical system enhances the performance of the unit by providing an anoxic condition. Anodes such as aluminum evenly release coagulation agents, which react with phosphorus compounds.

The abovementioned motives inspired the current study. These results can lead to a better understanding of the process and improve nutrient removals in the future.

1.3 Objective

The main objective of this study is to enhance nitrogen removal through electrokinetic control of redox conditions in a single wastewater reactor. This novel reactor was comprised of a layer of media for biofilm formation as well as suspended culture growth in aerobic and anoxic conditions.

Detailed objectives of the investigation were:

i) Comparative studies between activated sludge (AS) and media fixed-biofilm reactors (media reactor) in order to assess their response to nutrient removal;

ii) Finding the effect of media fixed-film on the nitrification/denitrification process;

iii) Assessing the nitrification capacity of an Integrated Fixed-film Activated Sludge reactor;

iv) Investigating simultaneous removal of nitrogen, phosphorous and carbons in this novel reactor;

v) Investigating the removal of nitrogen compounds from wastewater with elevated ammonia concentration.

Chapter II

Literature review

In order to meet Quebec and Canada's high effluent quality standards, nutrients removal (particularly ammonia) is a major concern (Government Canada, 2020). Nitrogen removal units operate on two main processes consisting of physicochemical and biological processes. Physicochemical processes remove nitrogen and are carried out by air stripping, ion exchange and oxidation, ammonia vacuum distillation, activated carbon adsorption, and electro-coagulation (EC) (Davis, 2013). Nitrification/denitrification and anaerobic ammonium oxidation (anammox) are the main biological nitrogen removal methods used in these systems.

The physicochemical process application is limited due to disadvantages such as scaling, additive chemical usage, and low-quality effluent. Similarly, EC is an efficient wastewater treatment process in the removal of colloids (Ibeid, 2011). However, it has only been limitedly applied in wastewater treatment due to its partial nutrient removal (such as ammonia), and difficulties it causes in the sludge treatment process. Consequently, biological nitrogen removal methods are vastly applied in wastewater treatment plants.

The overview of IFAS as an ammonium removal method is provided in this literature review. The electrical field was utilized in the EIFAS reactor to improve the performance of the IFAS reactor. Moreover, previous studies on membrane bioreactors (MBR) and submerged membrane electro-bioreactors (SMEBR) are summarized in this chapter. Since electrical current is applied in the

EIFAS reactor and the SMEBR, and IFAS and MBR systems are similar in the application of suspended culture.

2.1. Biological nitrogen removal

Biological nitrogen removal methods have evolved over the last three decades. Initially, nitrogen was removed by nitrification and denitrification processes in two separate rectors (Wu et al., 2007). This method requires a large area and has certain drawbacks, such as soluble microbial products, floc formation, and difficult sludge treatment. After which, membrane bioreactor and advanced anammox bioreactor were used to improve effectiveness of the process.

2.1.1 Nitrification/denitrification

Nitrogen in sewage is mostly in the form of ammonia and proteins. Protein gradually converts to ammonium due to biochemical reactions. Thus, the main objective of nitrogen elimination in wastewater treatment plants is ammonia removal; since it causes eutrophication and aquatic animal toxicity (Metcalf & Eddy, 2002). Nitrification bacteria grow and convert ammonia to nitrite under aerobic conditions which then oxidize to nitrates. Activity of these bacteria highly depends on the presence of oxygen and alkalinity. Nevertheless, complete nitrogen removal requires elimination of produced nitrates from the reactor. Anoxic conditions are essential in activation of anoxic bacteria which convert nitrates to nitrogen gas (Davis et al., 2013). Despite nitrifying bacteria, denitrifying bacteria complete a shorter life cycle, breed faster, and consume a greater load of organic carbon as a source of energy. Figure 2-1 shows the nitrogen cycle in nature. Each stage requires unique conditions and activity of a specific type of microorganisms.

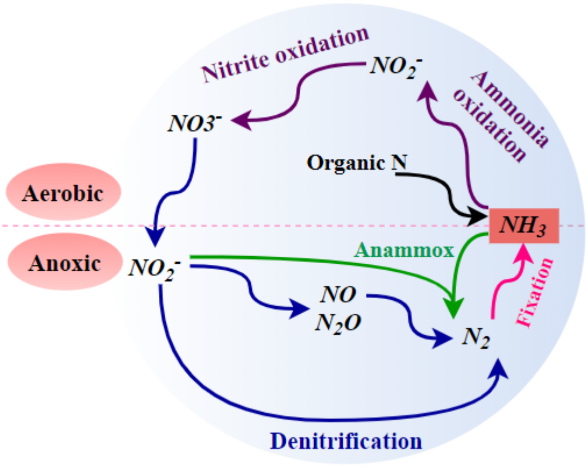


Figure 2-1 Nitrogen cycle in nature

Partial nitrification and anaerobic ammonium oxidation (anammox) processes took place in multiple zones of the bioreactor (Hosseinpour et al., 2019). Different dissolved oxygen levels were maintained in these zones and a combination of suspended and attached cultures was used in that study. Nitrite-free synthetic wastewater with high ammonium concentrations of 10-350 mg/L was treated in a multi-zone hybrid airlift bioreactor; 85.6% ammonium and 81.2% total nitrogen removal were achieved (Hosseinpour et al., 2019).

The biomass loss was compensated by anammox-containing sludge injection into the reactor to demonstrate a successful start-up. In the aerobic zone, the dissolved oxygen was between 0.9 mg/L and 1.2 mg/L. Hydraulic retention time (HRT) of the system varied from four to two days (Hosseinpour et al., 2019). The reactor's high HRT and anammox-containing sludge injection are disadvantages of that study in mainstream applications.

2.1.2 Anammox

Nitrification and denitrification processes require high energy to provide aerobic conditions as well as the need for an organic carbon source. On the other hand, anammox is an anaerobic bacteria, recognized for its high performance, environment-friendly behaviour, and low-cost ammonia removal in the presence of nitrite as an electron acceptor.

Anammox reduces the emission of greenhouse gases by 90%, given that CO_2 is used as a carbon-source instead of organic carbon and does not emit N_2O (Hu et al., 2010). Less sludge production, and no demand for aeration or organic carbon sources save about 60% of operational costs compared to conventional methods (Hu et al., 2010).

Although anammox can keep nitrogen removal performance at high salinities (30 mg/L NaCl) in ambient temperature (Yanga et al., 2010), changes in DO, PH, and temperature affect the performance of these bacteria. Moreover, low growth rate is an obstacle for vast applications in large-scale mainstream and sidestream.

At Concordia University, growth of anammox in different temperatures and in the presence of electrical systems has been investigated. The electrical current reduces oxygen that provides an anoxic condition and anammox growth in temperatures as low as 8 °C (Adam, 2019). In the same study, rapid growth and biomass accumulation were achieved in three months due to conditions provided by the electrical field (Adam, 2019).

2.2 Biological nitrogen removal processes and operation units

Biological nitrogen removal units require presence of microorganisms in the form of suspended culture in the reactor or attached culture on the surface of a medium. Each of these biological cultures is applied in different operating units. In section 2.2, a review of the previous study regarding different operating units is presented.

2.2.1 Suspended culture

2.2.1.1 Activated sludge

Activated sludge systems are mainly designed to degrade organic compounds in an aerobic tank. The carbon source is used as substrate for microorganisms in the form of sludge. Organic carbon compounds are presented in the form of Biochemical Oxygen Demand (BOD) or COD in lab measurements (Zhang et al., 2014). Four stages in the life of microorganisms are: lag time, exponential growth, stationary growth, and death; each of these stages can be divided into sub-stages. Some factors accelerate or delay each stage such as substrate availability, dissolved oxygen level, temperature, pH, and salinity (Davis, 2013).

Initially, the main purpose of activated sludge systems was to remove COD or BOD. Later, its application broadened to nitrogen removal as well. Nitroxide, nitrogen gas, nitrite, and nitrate are generated as by-products of this process. Another main issue of activated sludge systems is massive production of sludge, which requires additional operation units in order to be treated. Likewise, the removal of by-products generated in the nitrogen biodegradation process demands anaerobic/aerobic operation units (Cho et al., 2005). Any additional unit needs extra capital, requires more land, and has additional operation and maintenance costs.

A sequential anoxic/aerobic bioreactor (SA/AB) provides nitrification and denitrification conditions in a single reactor (Cho et al., 2005). The step-feed anoxic/oxic biological nitrogen removal process which distributes influent flow (Zhu et al., 2007), and internal flow recycling that helped to remove above 68% nitrogen from domestic wastewater are the examples of effort in improving ammonia removal in SA/AB system (Ahmed at al., 2006).

Although the design and performance of SA/AB have been enhanced during the last two decades, effluent quality and rates of influent treatment have not met all the requirements. Moreover, chemical additives and extra operation units are necessary to remove phosphorus compounds.

2.2.1.2 MBR

The effluent of activated sludge reactors requires secondary clarifiers and filtration to reach permissible total suspended solid levels. The membrane bioreactor eliminates the necessity of a secondary clarifier since it combines membrane filtration with the activated sludge process. Nowadays, membrane bioreactors (MBR) are widely applied in various industries such as wastewater and water treatment due to their better effluent quality and a smaller footprint. Membrane fouling remains one of the major drawbacks of this process.

Aerated membrane bioreactors can remove nitrogen by intermittent aeration of the reactor and adjusting the period of anoxic and aerobic phases in a cycle. The advantages of this method are rapid nitrification and a better denitrification process in high MLVSS concentrations without sludge deposition demand. In high SRT on average, 96% of TCOD, 100% SS, 83% of TN can be removed with an HRT of 8–15 hours (Yeom et al., 1999). Figure 2-2 shows a simple submerged (inside the tank) membrane in the aerobic reactor with air diffusers at the bottom of the tank. Another configuration for membrane reactors is called sidestream membrane (out of the tank).

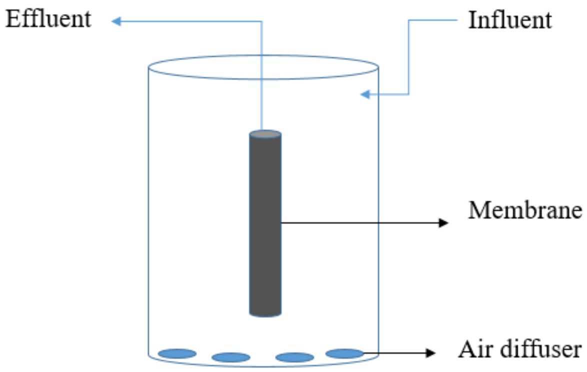


Figure 2-2 Aerated submerged membrane bioreactor

Suspended solids, colloids and solutes are the main sources of membrane fouling in wastewater treatment. Membrane fouling is also affected by operation conditions, sludge characteristics, wastewater composition and types of membranes. In most cases, fouling is irreversible. However, if activated sludge flocs and a cake layer of large particles deposit on the membrane surface, it can be reversible. Even though backwashing can remove reversible depositions, adsorption of dissolved organic material and solutes into membrane pores remains permanent (Ibeid et al., 2016).

Membrane fouling can be mitigated through intermittent aeration as well as changing of membrane surface properties to hydrophilic by introduction of polar groups (Yeom et al., 1999). Other technics such as changing the depth of the membrane module and using air for back-flushing are considered as physical methods to reduce fouling (Visvanathan et al., 1997). Moreover, the addition of adsorbent materials like powdered activated sludge (Kim et al., 2003), and optimizing the volatile suspended solids' concentrations (Ibeid et al., 2016) showed promising results.

MBR has promising COD removal and has been applied in wastewater treatment plants worldwide. However, it does not remove nutrients sufficiently and requires additional units to remove phosphorus and nitrates. To address the fouling problem in MBR, regular cleaning (physical or chemical) can be done. However, this solution leads to not only reactor efficiency reduction but also higher operational costs for chemicals and membrane replacements (Borea et al., 2016).

2.2.1.3 SMEBR

The combination of bioreactor with an electrical system is a novel technology to achieve high removal of nutrients (nitrogen and phosphorus) in wastewater. SMEBR is a hybrid system in which a complete mixed bioreactor comes with a submerged ultrafiltration or microfiltration membrane, as well as a system of electrodes. Maria Elektorowicz and Khalid Bani-Melhem registered a US patent regarding submerged membrane electro bioreactor (SMEBR) to enhance effluent quality, occupying less space, and to reduce membrane fouling (US patent n°12/553,680, 2009).

Membrane fouling is the main factor obstructing the wide application of membrane bioreactor (MBR) technology. Furthermore, enhanced biological phosphorous removal would be limited in MBR applications. A study on a SMEBR was conducted in order to decrease the fouling rate and increase performance in terms of phosphorus removal. Biological, membrane and electrochemical processes were combined in one operational SMEBR unit. It achieved 52% reduction in membrane fouling and more than 98% efficiency of PO_4-P removal (Bani-Melhem et al., 2009).

Another study discovered the relation between membrane fouling and other operation conditions which affect sludge characteristics; voltage gradient, Current Density (CD) and exposure mode.

The medium current density between 15 and 25 A/m^2 considerably improves sludge characteristics; better dewaterability, high removal of soluble microbial products (SMP) and organic colloids which reduces membrane fouling. Nutrient removal (up to 97% for nitrogen, 95% for carbon, and 99% for phosphorus) demonstrates promising results for the SMEBR hybrid system in wastewater treatment (Ibeid, 2011).

To be more specific, electro-coagulation and biodegradation take place between the anode and cathode area, whereas membrane filtration and biodegradation occur between the cathode and the membrane module. When 5 minutes ON and 15 minutes OFF were used intermittently as a pattern for current exposure times, optimum results for nutrient removal were achieved (Ibeid, 2011).

In a novel research, anammox bacteria were applied in an aerated submerged electro bioreactor. This was done to use aerobic conditions for nitrifiers and anoxic conditions for denitrifiers and anammox bacteria in the same reactor. In steady-state operations, the removal efficiency of ammonia, nitrate, phosphorus and COD in the SMEBR-Anammox were recorded as 97%, 99.95%, 99.91% and 99.87%, respectively (Hosseini, 2016).

The enhanced electro bioreactor was subjected to a numerical modelling study. The four-stage hybrid model consisted of hydraulic, activated sludge, electrocoagulation, and membrane filtration model conducted in MATLAB R2011b. The influent characteristics derived from lab experiments on raw medium-strength wastewater in real operation conditions. The current density between aluminum and stainless steel electrodes was 15 A/m^2 with intervals of 5 minutes ON and 15 minutes OFF.

In this study, experimental results were similar to steady-state modelling results. The modelling of removal efficiencies for COD, TP, TN, Ni, Fe, and Cr were 99, 99.3, 90.6, 79.3, 88.6, and 79.8% respectively. Besides, experimental results demonstrated removal efficiencies of mentioned elements as 96.6, 99.3, 90.6, 86.6, 86.1, and 79.3% respectively. From the modelling results, the inserted electrodes improved removal efficiencies from raw influent wastewater. Applying electrokinetics in wastewater decreases the plant footprint and fouling problems (Giwa et al., 2015).

Energy consumption in conventional wastewater treatment plants is quite high because of need of several operation units to remove nitrogen-containing compounds. The low current density between electrodes and limited aeration in the nitrification process decreases the overall cost of SMEBR. Removing nutrients and improving sludge properties in a single reactor are major advantages of this system. Operation conditions of this system are flexible and are related to the shape and size of the reactor. Furthermore, when the system faces unexpected shocks, process recovery is faster than when it happens in the MBR system.

Figure 2-3 shows a SMEBR in which a centred submerged membrane is surrounded by a perforated cathode and anode in a completely mixed activated sludge reactor. A constant current density is employed to the system by these electrodes and proper aeration is injected. Therefore, diffusers are equally distributed in the system at the bottom of the reactor in order to provide mixing of liquor and electron acceptors to microorganisms.

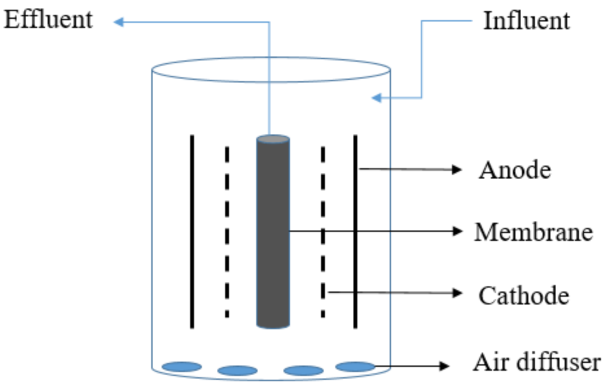


Figure 2-3 Aerated submerged membrane electro-bioreactor

2.2.2 Attached culture

Attached culture in wastewater treatment has been applied for centuries under the form of a trickling filter. This process consisted of a bed of coarse material, such as stones, slats, or recently more plastic material (media). In wastewater treatment, the process of biofilm growth on media surface is called attached growth. Conventional trickling filters were used with a bed of stones, 1 to 3 meters deep, through which the wastewater was filtrated (Davis et al., 2013).

Another method of applying attached culture is the Rotating Biological Contactor (RBC). It had a sequence of narrowly spaced discs with a diameter of 3 to 3.5 meters attached to a horizontal shaft. While about one-half of its surface area was immersed in wastewater, it rotated (Davis et al., 2013). Gradually, with constant contact of discs to wastewater, a layer of biofilm of 1 to 3 millimetres would form.

The other type of attached culture growth is Integrated Fixed-film Activated Sludge (IFAS). It is a hybrid reactor consisting of both suspended culture (activated sludge) and attached culture (fixed-film). The properties of media vary from sponge, polyurethane foam (PUF), and plastic carriers, to polyester fabric.

For instance, a sponge tray is a simple approach to increase the efficiency of an attached culture bioreactor in terms of both organic and nutrient removal efficiencies. The type and shape of the sponge and the designated slope of the tray are key parameters in a successful design (Ngo et al., 2007).

In the past two decades, the application of moving bed biofilm reactors (MBBR) in wastewater treatment has increased. In most cases, a polyethylene media with a high surface area (250–515 m^2/m^3) was added to the bioreactor to fill about 30 to 60 percent of the reactor volume. The advantage of this media is that it floats on the surface and stays in contact with both air and wastewater.

Sponge modified bio-carrier reactors (S-MBBR) can increase the efficiency of the MBBR system. Also, it reduces membrane fouling in a hybrid MBBR–MBR system. Sponge modified bio-carriers can reduce the amount of soluble microbial products (SMP) in mixed liquor and extracellular polymeric substances in activated sludge systems. Thus, it alleviates cake layering and pore-blocking resistance of the membrane. S-MBBR has a notably better NH_4-N and COD removal than the MBBR system. At an HRT of 12hr, S-MBBR removes NH_4-N and COD by 83% and 95% respectively, while MBBR was only able to remove 72% and 91%, respectively (Deng et al., 2016).

Applications and types of media are varied. For example:

1. All types of industrial and domestic wastewater.
2. Organic nitrogen removal.
3. Upgrade of existing or new plants.

IFAS combines the activated sludge process and biofilm in a treatment unit and enhances the capacity of BOD/COD removal. Furthermore, it has a small footprint, strong denitrification and dephosphorization ability. It enhances the activated sludge process without need for a new unit.

2.3 Phosphorus removal

Phosphorus in water over the allowable amounts is considered a pollutant in water bodies such as lakes, rivers, reservoirs, and streams (Muscutt et al., 1996). Phosphorus enrichment causes Eutrophication in aquatic systems and algal blooms. The growths of algae adversely affect water quality, impair downstream water treatment processes and restrict recreational activities (Clark et al., 1997).

Two major phosphorus removal mechanisms are the biological process and coagulation. The biological phosphorus removal requires a separate anaerobic tank followed by an aerobic tank (Davis et al., 2013). The coagulation process is a Physico-chemical process. Physical settlement occurs in the settlement tank after chemical reactions with coagulants.

Another effective process that can be applied in the industry is the Electrokinetic or Electrocoagulation process. An electro-bioreactor such as SMEBR will release Al^{3+} (coagulation agent) due to oxidation of the aluminum anode (Hasan et al., 2014). The aluminum ions react with phosphorus compounds, especially orthophosphates which are soluble compounds.

Details of the reactions are presented below (Equations 2-1 to 2-3).

$3Al^{3+} + 2PO_4^{3-} + 3H_2O \rightarrow (AlOH)_3\ (PO_4)_2 + 3H^+$ Equation (2-1)

$Al^{3+} + PO4^{3-} \rightarrow AlPO_4$ Equation (2-2)

$Al^{3+} + 3H_2O \rightarrow Al(OH)_3 + 3H^+$ Equation (2-3)

Therefore, PO_4^{3-} precipitates in the form of $(AlOH)_3(PO_4)_2$ and $AlPO_4$ or it is adsorbed by $Al(OH)_3$ which is a strong adsorption agent (Wei et al., 2009).

As was previously mentioned, the biological treatment of phosphorus requires more complex conditions than biological nitrogen removal. Biological nitrogen removal can occur in a single reactor, but phosphorus cannot be removed without an additional reactor (Jianlong et al., 2008).

In this study, performance of the Electro-Integrated Fixed-film Activated Sludge reactor is verified where nitrification and denitrification processes take place in a single vessel. Considering nitrifying and denitrifying bacteria are responsible for nitrogen removal, it is essential to study the optimum living conditions of each microbial culture.

Chapter III

Methodology

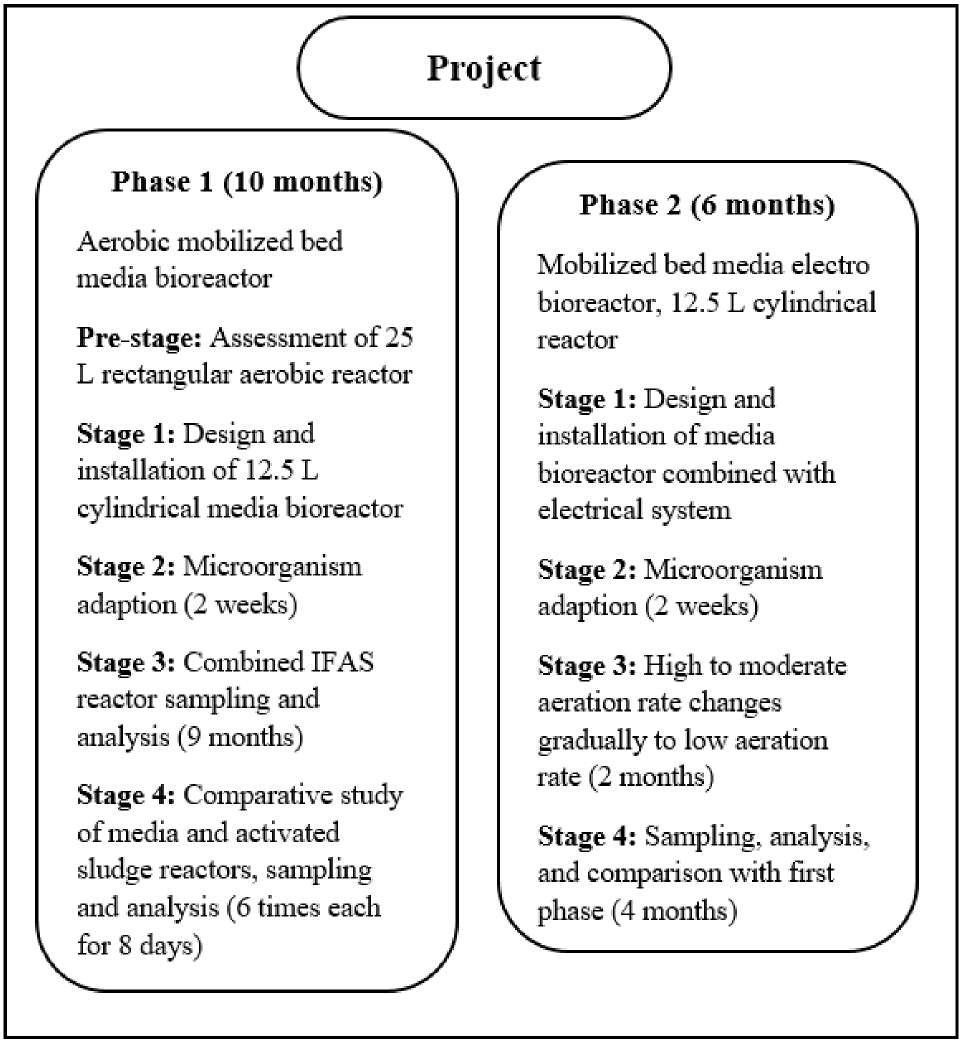
This experimental study was done over a period of 16 months, consisting of setup preparation, installation and experimental stages. The study had two phases with different operation conditions. The characteristics of the synthetic wastewater and HRT remained constant throughout the study. The nutrients removal rate, mainly ammonia and nitrate, was assessed in each phase.

The concentration of parameters such as DO, ammonia-nitrogen, nitrate-nitrogen, and COD was continuously measured. Moreover, the variations of oxidation-reduction potential (ORP), total Kjeldahl nitrogen (TKN), current input, phosphate removal, total suspended solid (TSS), mixed-liquor volatile suspended solid (MLVSS), nitrite-nitrogen, and total nitrogen (TN) were recorded. These parameters were recorded based on importance of corresponding parameters in that phase.

The main objective of the study was to assess the ammonia-nitrogen (NH_3^--N) removal as well as the removal of nitrate-nitrogen (NO_3^--N) which is a by-product of the nitrification process. The two main substrates, ammonia sulphate and dextrose (D-glucose), were added to synthetic wastewater to provide the required carbon and nitrogen source for microorganisms.

The synthetic mix had a constant ratio of compounds throughout the study, except for ammonia sulphate which was increased while system response to it was being evaluated. Hence, the comparison is robust and valid. Ammonia-nitrogen (NH_3^--N), nitrate-nitrogen (NO_3^--N), total Kjeldahl nitrogen (TKN), and total nitrogen (TN) were the indicators of nitrogen in the system and were all measured during the study. In addition, orthophosphate (PO_4^{3-}) and phosphate ions (PO_4^{3}) were the major forms of phosphorous.

Table 3-1 Overview of the project



Project

Phase 1 (10 months)

Aerobic mobilized bed media bioreactor

Pre-stage: Assessment of 25 L rectangular aerobic reactor

Stage 1: Design and installation of 12.5 L cylindrical media bioreactor

Stage 2: Microorganism adaption (2 weeks)

Stage 3: Combined IFAS reactor sampling and analysis (9 months)

Stage 4: Comparative study of media and activated sludge reactors, sampling and analysis (6 times each for 8 days)

Phase 2 (6 months)

Mobilized bed media electro bioreactor, 12.5 L cylindrical reactor

Stage 1: Design and installation of media bioreactor combined with electrical system

Stage 2: Microorganism adaption (2 weeks)

Stage 3: High to moderate aeration rate changes gradually to low aeration rate (2 months)

Stage 4: Sampling, analysis, and comparison with first phase (4 months)

3.1 Synthetic wastewater

3.1.1 Introduction

The focus of this study was to investigate the nutrient removal in a hybrid electrical system and a media reactor in different operation conditions. Therefore, a laboratory-scale system was designed to treat synthetic wastewater. The sources of nutrients utilized to prepare synthetic wastewater are listed in table 3-2. The synthetic wastewater was prepared with all necessary nutrients needed for the growth of the type of microorganism targeted. Synthetic wastewater was prepared every 4 days. The chemicals were weighted accurately to a thousandth of a gram and well mixed to a concentrated solution on a hot-plate. The concentrated solution was then added to 100 litres of tap water. Before making fresh synthetic wastewater, the containers were cleaned and remaining wastewater was thrown away in order to not affect the influent concentrations.

Table 3-2 Synthetic wastewater components

Component	Concentration(mg/L)
Ammonium sulphate	**200**
Potassium phosphate	**37**
Magnesium sulphate	**40**
Manganese sulphate	**4.5**
Iron sulphate	**0.4**
Calcium chloride	**4**
Potassium chloride	**25**
Sodium bicarbonate	**25**
Glucose	**360**

3.1.2 Source of nutrients

The two main nutrients responsible for assessing the quality of wastewater are nitrogen and phosphorus and untreated wastewater can cause many issues in the environment. In this study, a specific list of chemical compounds was used to prepare synthetic wastewater which has all required nutrients for microorganisms.

The ratio of COD:N:P in the synthetic wastewater was approximately 40:4:1. This ratio was used in order to provide a similar nutrient concentration to domestic wastewater. Moreover, at some points of the investigation, the ammonia-nitrogen concentration was increased to evaluate the performance of the system in similar conditions to industrial wastewater treatment processes.

Ammonia-nitrogen and proteins are common types of nitrogen in the wastewater. Proteins convert to ammonia in some biochemical processes. Thus, ammonia-nitrogen is the most common nitrogen source in wastewater. In this study, ammonia sulphate was the source of nitrogen in the influent. Due to dissociation, ammonium cations (NH_4^+) are the major form of nitrogen in the synthetic wastewater; minute amounts of nitrites and nitrates are also present.

The balance of nitrogen compounds changes in the reactor due to biodegradation. Thus, monitoring of all nitrogen species (including nitrites and nitrates) is necessary for evaluation of the effluent quality. Observation of ammonia-nitrogen, nitrite and nitrate levels in both influent and effluent show the level of nitrification and denitrification in the IFAS and EIFAS.

For the duration of this study, in every feeding preparation, 20 grams of ammonium sulphate was dissolved in 100 litres of water. This would result in a synthetic wastewater with approximately 40 mg/L of ammonia-nitrogen. Several months after keeping the system at constant influent preparation concentration, the amount of ammonium sulphate was increased in order to evaluate the maximum capacity of ammonia removal and the system's response to overload and shocks. Thus, ammonia-nitrogen concentration amounts of up to 65 mg/L were recorded as the influent nitrogen concentration.

Another source of nutrient was phosphorus which was provided in the form of potassium phosphate (KH_2PO_4). An amount of 3.7 g of potassium phosphate was added to 100 L of water in order to have 15 mg/L to 17 mg/L of PO_4^{3-} concentration. The concentration of phosphate remained the same throughout the study.

The variation of phosphorous concentration in the influent may be a result of incomplete dissolution of phosphorus sources during the preparation of wastewater and/or settling and uptake of phosphorus sources in the influent tank over time. The variance was recognized to be insignificant and therefore negligible.

An amount of 36 g of D-glucose (organic carbon) was added to every 100 L of synthetic wastewater. The influent COD concentration was between 350 mg/L and 450 mg/L throughout the study. The reason for this variance was COD degradation and settling potential.

3.2 Experimental setup

This study was conducted in two phases; Integrated Fixed-film Activated Sludge and Electro-Integrated Fixed-film Activated Sludge. The experiment was conducted in a cylindrical reactor; the effective volume was 12.5 L. The biofilm carrier (media) in this study was applied in both phases.

The application of moving bed media is combined with biological wastewater treatment in advanced wastewater treatment methods used for industrial and municipal wastewater. It increases the capacity and efficiency of existing wastewater treatment plants considerably, as well as reducing the new plant footprint.

In this study, plastic media with a nominal diameter of 25 mm and a height of 12 mm was applied in both phases. The specific density, specific surface area, and porosity were 0.96, 500 m^2/m^3, and 90%, respectively. The media occupied 30% of the reactor's volume, which was an optimum ratio for this system. Figure 3-1 shows the plastic media used for biofilm building. Characteristics of the media bio-carrier are provided in table 3-3 (Plastic Biofilter, 2020).

Figure 3-1 Image of plastic media used in this study

Table 3-3 Media bio-carrier characteristics (Plastic Biofilter, 2020)

Characteristics	**value**
Dimension (mm)	**25 diameter × 12 height**
Density (kg/m^3)	**960-980**
Porosity	**$\geq$ 90%**
Surface area (m^2/m^3)	**> 500**
Hole numbers	**19**
Life time (year)	**> 15**

At the beginning of the experiment, an adequate amount of dissolved oxygen (DO) was injected into the system through the air stones at the bottom of the reactor. It provided the desired conditions required for the growth of nitrifying aerobic bacteria in the activated sludge as well as on the surface of media.

3.2.1 Activated sludge

At the beginning of the study, 12.5 L of secondary activated sludge was taken from Ville St Catherine wastewater treatment plant in Quebec and injected into the reactor. The concentration of TSS in the original sludge was approximately 1000 mg/L. This concentration showed that sludge had a diluted quality at the beginning of the study; this trend continued during the first phase. In the second phase, solid concentration increased due to better aeration control. Therefore, microorganisms would stay within the system and were not washed out as much as in the previous phase.

3.2.2 Synthetic wastewater

The applied synthetic wastewater had similar components to domestic wastewater such as the carbon and nutrient sources. Nitrogen and phosphorus were the two main nutrient components of the wastewater used, as presented in table 3-2. All but one component were kept at a constant ratio throughout the study; ammonia sulphate was increased at certain intervals. As it was mentioned before, nitrogen input was increased in order to evaluate the response of the system to a shock, as well as to find the maximum capacity of the system to remove ammonia.

3.2.3 Integrated Fixed-film Activated sludge reactor (IFAS) (phase I)

The cylindrical tank was designed based on its proven performance in studies on activated sludge (Ibeid, 2011; Gao, 2014). Overall, 30% of the reactor was filled up with media. The rest of the tank was filled with sludge (a combination of synthetic wastewater and activated sludge for inoculation).

Several air-stones injected air into the system, providing mixing of liquor and floating of media. Daily manual physical mixing helped the media bioreactor perform as a complete mix reactor. Synthetic wastewater was pumped into the system at a rate of 25 L/day and effluent was discharged at a similar rate. The effective reactor volume was 12.5 L; HRT of the system was 12 hr. The purpose of this phase was to reach high ammonia oxidation levels; adequate aeration was provided to the reactor in a range of 5 mg/L to 8 mg/L. Figure 3-2 shows the laboratory setup and schema for the Integrated Fixed-film Activated Sludge reactor in phase I.

Once treatment was complete, the remaining nutrient concentrations of the supernatant effluent were measured by passing it through a filter with a pore size of 0.45µm (Fisher Scientific, 2019). After which it was compared to the nutrient concentration in the influent in order to assess removal efficiencies. Since breeding and growth of nitrifying bacteria require time, this phase (phase I) lasted ten months during which results were recorded and analyzed.

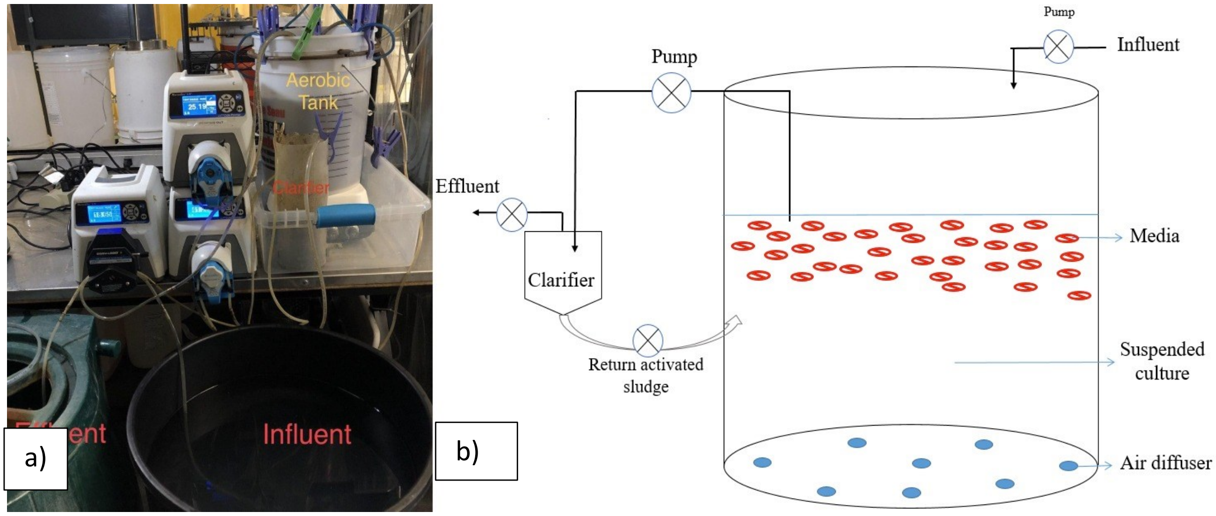


Figure 3-2 Integrated Fixed-film Activated Sludge a) Image of laboratory setup picture b) Schema of the reactor

3.2.3.1 Comparative study of attached culture and suspended culture

The IFAS is a combination of the activated sludge system (AS) and the mobilized bed biofilm (media reactor). AS and media that form the IFAS were separated in to two reactors in order to assess the capacity of their treatments separately; particularly nitrification capacity. These comparative studies were conducted after 6, 10, 13, 15, 21, and 29 weeks following the start of the experimental study in phase I for a period of 8 days. To reach a robust assessment, the same operation conditions were applied to separated reactors similar to IFAS; such as synthetic wastewater, DO concentration, and HRT.

The media reactor contained only tap water on the first day. For this reason, the media attached biofilm was the only source of treatment in the media reactor. Measurement of removed nutrients demonstrated the bio-carrier capacity to remove ammonia and COD. Feeding of the conventional activated sludge reactor and the media reactor was done with the same prepared synthetic wastewater and effluent qualities were recorded for comparison. The schema of comparative reactors, i.e., the media reactor and activated sludge reactor are shown in figure 3-3.

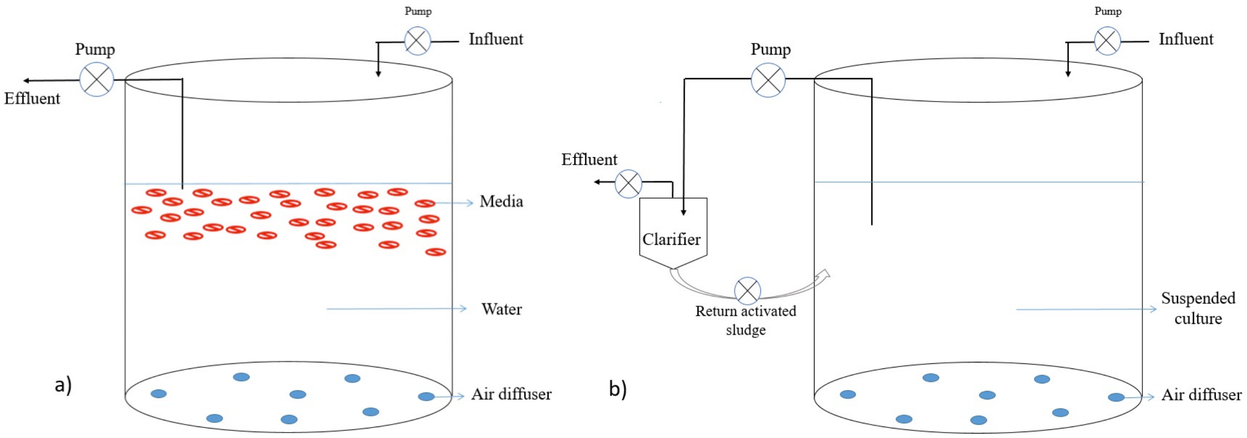


Figure 3-3 Comparative schemas of two reactors: a) Media reactor b) Activated sludge reactor

3.2.4 Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS) (phase II)

The EIFAS electrical system consisted of two cylindrical perforated electrodes (aluminum anode and stainless-steel cathode) and a DC power supply (controlled by a timer) that provided intermittent current. Porosity of the anode was 40%. Distributed air diffusers at the bottom of the reactor provided an adequate aeration as well as mixing of the liquor. Moreover, daily manual physical mixing helped the EIFAS to perform as a complete mix reactor.

Figure 3-4 shows a schema of the second phase EIFAS of this study. The Electrical current was conducted by a perforated aluminum anode with a diameter and height of 20 cm surrounding a cathode with the same height and diameter of 10 cm. As is shown in figure 3-4, the media was situated in the outer zone of the reactor between the anode and the reactor wall; some media were placed inside the cathode zone. The volume of the outer zone, which is the space between the anode and wall of the reactor, was 7.2 L and plastic media occupied more than 80% of this space. Because of this, the plastic media was not moving in this area. Like that, inside the cathode was jam-packed with plastic media. Very few media was placed in the zone between anode and cathode in order to not create a shortcut in the electrical circuit by media bridge. A summary of EIFAS design parameters is given in table 3-4.

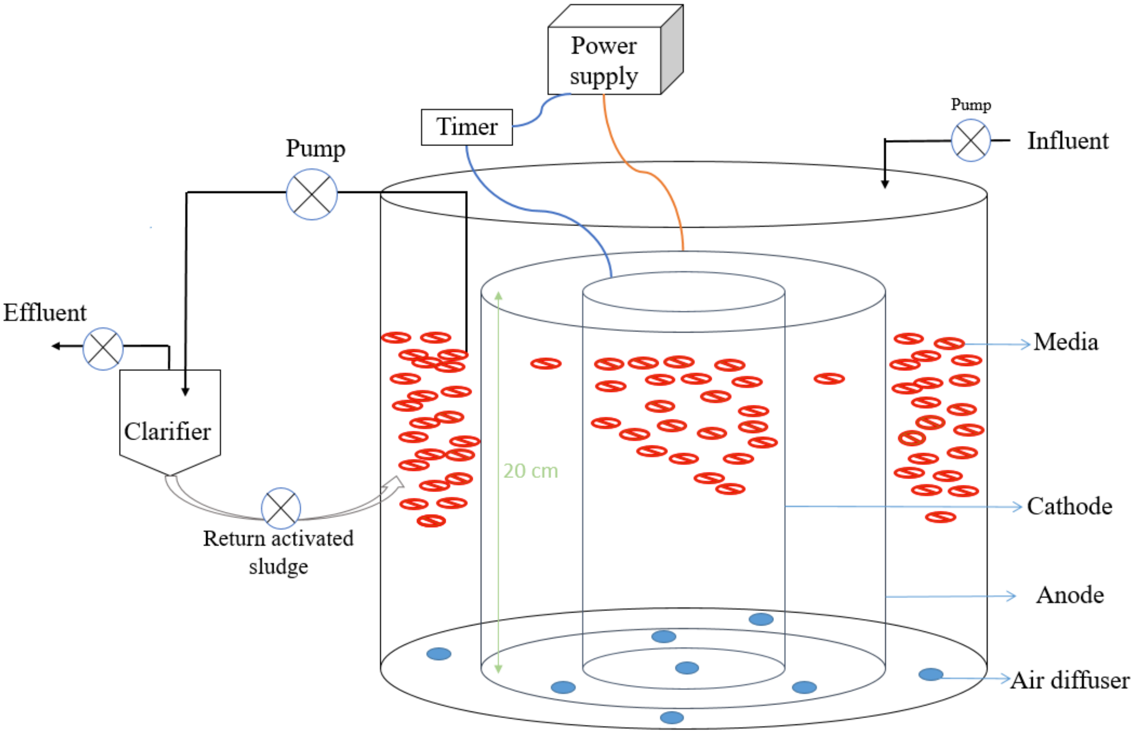


Figure 3-4 Schema of enhanced Electro-Integrated Fixed-film Activated Sludge reactor

Considering that the anode material would be released to the liquor in the electrochemical process, selection of the anode material is important. In these systems, phosphorus removal depends on anions released from the anode. High valence ions, affordable price, and availability are adequate properties of aluminum to be chosen as industrial and lab-scale anodes.

Table 3-4 Design parameters of the Electro-Integrated Fixed-film Activated Sludge reactor

Parameter	value
Effective reactor volume (L)	**12.5**
Submerged height of electrode (cm)	**17**
Anode diameter (cm)	**20**
Cathode diameter (cm)	**10**
Anode surface porosity (Φ) %	**40**
Effective anode surface area (cm^2)	**640**

3.3 Operation conditions

Hypotheses, system design, and monitoring the operation conditions are the key components to reach the expected results. Operation conditions such as flow rate, current density and biomass concentration are all considered dynamic and static features of the system which need to be controlled. For a continuous flow reactor, continues monitoring and maintenance are crucial in order to keep the system running properly. Cleaning the connecting tubes, media surface and influent tank, as well as control of flowrate and voltage, are examples of monitoring and maintenances required. In the following sections, details of operation conditions will be discussed.

3.3.1 HRT and SRT

Hydraulic retention time (HRT) was 12 h in order to simulate actual HRTs applied in industrial reactors. Furthermore, this HRT value is acceptable according to the previous study, where it was recommended to set the HRT between 6 hours and 15 hours (Hasan et al., 2014).

Solid retention time (SRT) indicates the period biomass will remain in the reactor. Sludge concentration and quantity depend on the SRT. Therefore, it needs to be adapted based on the type of microorganisms expecting to grow, growth rate, decay rate and overall sludge properties. In this study, SRT was mostly kept on 20 days according to previous studies.

The volume of reactor was 12.5 liters and sludge occupied 70 percent of it. SRT of twenty days was controlled by removing three litres of bottom sludge per week. High SRT allows biomass concentration; Therefore SRT was kept higher than 20 days in the adaptation stage in phase I. Moreover, in the case of overflow or other unexpected accidents, the SRT was modified in order to keep the appropriate sludge concentration in the system.

3.3.2 Current density

The point of using an electrical field is to control redox potential in a bioreactor where denitrifying bacteria can grow in the same tank as denitrifying bacteria. This happens when electrochemical reactions cause "oxygen consumption" and create anoxic conditions. The operation mode was adjusted in this study to response the required redox conditions. Subsequently, an intermittent DC field was applied.

Thus, the optimal electrical exposure mode applied in this study was 20 min OFF and 5 min ON, in accordance with previous investigations (Ibeid et al., 2013). In this study, the voltage was adjusted by the use of a DC power supply. This was done in order to maintain the level of current density between 12 A/m^2 and 15 A/m^2. The summary of the operation conditions for both phases are presented in table 3-5. In order to calculate current density, the effective anode surface and current are needed. Current density and effective anode surface were calculated by equations 3-1 and 3-2, respectively.

Current density (CD) = I / Effective anode surface Equation (3-1)

$\text{Effective anode surface} = 2\pi Rh\,(1-\Phi) = 20\,\pi \times 17 \times 0.6 = 640.88\ \text{cm}^2$ Equation (3-2)

Table 3-5 Operation conditions detail for both phases

Condition	IFAS	EIFAS
Average sludge concentration (mg/L)	3000-4000	5000-6000
Sludge volume (L)	12.5	12.5
Weekly sludge take out (L)	300	400-500
DO (mg/L)	6-8	1-3
HRT (hr)	12	12
SRT (days)	10-20	20
Current density (A/m^2)	-	12-15

3.4 Measurement and analyses

3.4.1 Measurement goals and methods

There were two types of measurement throughout the study: i) regular daily measurements, and ii) unsystematic measurements. Both types not only showed progress of the experimental study, but also showed where problems were occurring in the reactor. Regular measurements consisted of ammonia-nitrogen, nitrite-nitrogen, nitrate-nitrogen and DO concentrations and ORP values.

Unsystematic measurements quantified treatment results as well as operation conditions. Because the results were relatively stable or the importance of a particular indicator was not high, daily measurements were not necessary. Non-frequent unsystematic measurements were conducted every week or occasionally, depending on the measurement objectives. These measurements included those done for orthophosphate, COD, total nitrogen, TKN, and total suspended solids.

The measurement methods consisted electrode probe, TNT Hach kits (Product- TNT plus^{TM} Chemirtries), and Ion Chromatography (IC). Each of the above-mentioned methods was utilized to measure a specific parameter or used to compare the results of two methods.

In 2019, Metrohm IC machine (530 Compact IC Flex) was purchased with two sensitive columns used to measure anions and cations (Agilent OpenLab CDS). For sample preparation, microfiltration was required and the device needed regular calibration to ensure accurate results; this method was the most reliable. This device could run multiple samples when ion concentrations were in the same range. Nevertheless, column changing, sample preparation, device calibration, and results interpretation are challenges of using the IC.

In this study, the probes were utilized to measure DO and ORP, and the IC was used to measure ammonia, total phosphorus, nitrate, and nitrite concentrations. TNT Hach test measurements were used for ammonia-nitrogen, nitrate-nitrogen, orthophosphate, total nitrogen, TKN, and COD concentrations.

3.4.2 Dissolved oxygen concentration (DO)

Oxygen concentrations can create aerobic and anoxic conditions which can cause nitrification and denitrification processes in a bioreactor. Therefore, it is one of the most significant parameters considered in the study due to its role in nutrient removal; especially nitrogen.

In this investigation, DO measurements were conducted with the use of RDO optical dissolved oxygen sensors connected to a portable DO/RDO meter (Orion Star A223). At least three points of the reactor were measured for DO values daily. It would take at least 5 minutes for the probe to stabilize and show accurate values. Maximum, minimum, and average DO levels were reported to find a DO trend variation.

DO level was one of the variable operation conditions that highly affected treatment results. In phase I, a sufficient DO of 5 mg/L to 8 mg/L was provided in order to allow sludge and attached biofilm to operate in optimum nitrification reaction conditions. In phase II, to balance the concentration of ammonia-nitrogen and nitrate-nitrogen, the level of DO was kept between 1 mg/L and 3 mg/L to provide anoxic conditions.

DO levels are optimum if total nitrogen removals, including both ammonia and nitrates, reach the maximum allowable amounts. Removal results in the IFAS and EIFAS reactors were compared in order to assess the improvement of treatment methods. In the EIFAS reactor, DO concentration decreased during ON mode, especially in the area between the two electrodes, due to oxygen oxidizing. This means that the oxidization process would be slow, while denitrification process is sped up. The length of time when current was ON or OFF, directly controlled the duration of each reaction and DO concentration. The ON mode was 5 minutes versus 20 minutes OFF mode, since denitrifying bacteria breed faster than nitrifying bacteria. This ratio was applied based on previous studies (Ibeid et al., 2013).

3.4.3 Oxidation/reduction potential (ORP)

ORP is another indicator of nitrification and denitrification processes, which is directly related to the DO level. During this study, ORP was measured by using an electrode connected to the AB200 bench meter (Fisher Scientific). During measurements, a minimum of three readings were conducted in order to assure accurate results. Unfortunately, the DO probe was not working during the second phase, and OPR values were recorded for tracking the levels of oxygen within the system as well as oxidation/reduction state.

Theoretically, when current is applied, DO concentration would declines, and the system is in the heterotrophic phase (denitrification). In this phase, organic carbon sources such as glucose are consumed by denitrifiers and converted to nitrate gas (N_2). Moreover, ORP should be between 50 mV and -150 mV. When the current is OFF, oxygen oxidation is interrupted and DO concentration increases. At this time, the reactor is in the autotrophic phase (nitrification). Inorganic carbon sources and alkalinity are needed in this process. ORP is usually more than 100 mV in this stage.

During the second phase of this study, ORP was monitored and aeration distribution was changed in order to reach ideal ORP values. There were some incidents where the ORP values were out of this range and it was necessary to change the aeration manually.

3.4.4 Current density

The current applied to the EIFAS reactor is one of the essential parameters which needs to be controlled. The change of current input directly affects biological activity and nutrient removal efficiency results (Hasan et al., 2014).

The DC field affect Diffuse Double Layer (DDL) of colloidal sludge particles which flocculate and provide a large sorption area. Moreover, the anode oxidation may oxidize organic compounds to make them more bioavailable. The positive effect of the electrical field on sludge includes: pH control, decrease membrane fouling and deposition on electrodes. (Hasan et al., 2014).

Sludge conductivity can change over time due to sludge property alteration, passivation corrosion of electrodes, and sludge dumping. Therefore, monitoring the current on a daily basis is a necessity. The current density is also influenced by both the design of the reactor, particularly the anode, and the current input from the power supply.

In section 3.3.2, current density calculation details are explained. In this study, optimum current density was kept between 12 and 15 A/m^2. The current values in this reactor were between 0.77 A and 0.96 A (equation 3-3 to 3-5). Nevertheless, because of some accidents and changes in the system, some of the current measurements were out of range.

$I = CD \times \text{effective anode surface}$ Equation (3-3)

$12 \times 0.064 < I < 15 \times 0.064$ Equation (3-4)

$0.768 < I < 0.96$ Equation (3-5)

Table 3-6 Current density for each level of current input

Voltage (V)	Current (A)	Current Density (A/m^2)
13	**1**	**15.63**
12	**0.9**	**14.06**
10	**0.8**	**12.5**
8	**0.7**	**10.94**

3.4.5 TSS and MLVSS

Standard TSS and MLVSS test procedures include three stages (Zhang et al., 2014). First, a certain volume of the liquor is sampled from a complete-mixed reactor; for example, a 10 ml sample was taken. After which it is passed through a clean, dry, and heat resistant filter paper. Secondly, the sample is dried in an oven and a furnace in order to measure suspended solid concentrations.

For TSS measurements, the sample is dried at 105°C until all of the water evaporates and no further mass change occurs. It normally takes between 45 minutes and 12 hours depending on the sample type (equation 3-6).

After the total suspended solids value is determined a volatile suspended solids (VSS) test may be performed in order to determine the concentration of volatile suspended solids in an aeration stabilization basin system. Volatile suspended solids data is critical in determining the operational behavior and biological concentration throughout the system. The filter used for total suspended solids (TSS) testing is ignited at 550 °C for 30 minutes. The dried sample must stay in the desiccator until it cools down. At this point, MLVSS is calculated as the mass loss on ignition of the solids represents the volatile solids in the sample (equation 3-7).

Regular filter papers can withstand temperatures of up to 105°C, therefore it used for the TSS test. However, in order to avoid burning of regular filter papers at 550°C and a glass fibre paper is required for the MLVSS test.

$$\text{TSS (mg/L)} = \frac{\text{Mass of dried sample at 105°C – Mass of filter paper}}{\text{Volum of sample}} \qquad \text{Equation (3-6)}$$

$$\text{MLVSS (mg/L)} = \frac{\text{Mass of dried sample at 105°C– Mass of dried sample at 550°C}}{\text{Volum of sample}} \qquad \text{Equation (3-7)}$$

TSS and MLVSS are indicators of the concentration and volatile fraction of sludge. These two parameters were necessarily measured at the beginning of the study. A trend of TSS and MLVSS measurements showed changes in sludge quality, sludge concentration, and reaction conditions in the reactor.

The concentration of suspended solids in each stage changed in the first phase. Due to aeration in the bioreactor and absence of a submerged membrane, biomass would be washed out easily. In phase II, lower aeration would create a less turbulent system. The electrokinetic phenomena resulted in formation of bigger particles/flocs, which are more likely to remain in the reactor. Because of this, extra sludge was added to the system when biomass concentration was less than the amount required in the first phase; it was not required for the second phase.

3.4.6 Nitrogen concentration

Nitrogen in wastewater influent and effluent exists in the form of ammonia, nitrites, and nitrates. Ammonia-nitrogen and nitrate-nitrogen concentrations are presented in separate comparative graphs in chapter IV. The analysis of ammonia removal and nitrate concentration in relation to DO and ORP levels gives a better understanding of the level of nitrification and denitrification occurring in the reactor.

Furthermore, the results of reactor efficiencies in EIFAS and IFAS are compared to show the effect of the electrical field on nutrient removal. The optimum rate of nitrogen removal is stated as the conclusion of this study.

3.4.6.1 Ammonia-nitrogen

Since different forms of nitrogen convert to ammonia in biochemical reactions in raw sludge, ammonia is the main source of nitrogen pollution in raw sewage and industrial waste. Ammonia-nitrogen was measured in this study using two techniques. In phase I, measurements were done using TNT Hach kits and the IC machine. TNT 832 kits were used for a range of 2-47 mg NH_3^--N/L and the IC was used when kits were not available. Unfortunately, results of the IC became unreliable due to technical issues over time, and the TNT Hach test was the only method of measurement. In some cases, three samples were taken to compare and guarantee the accuracy of the results.

3.4.6.2 Nitrate-nitrogen

Nitrate is one of the main forms of nitrogen compounds and it is produced due to ammonia oxidation in the nitrification process when. Nitrate is generated in natural environments and biological wastewater treatment plants.

In the first phase of the study, the IC machine was used to measure nitrate concentrations. For the same reason as ammonia-nitrogen, unfortunately over time results of the IC became unreliable due to technical issues, and TNT Hach test was the only method of measurement used. In both phases, TNT Hach tests were used to measure nitrate-nitrogen concentrations; TNT 835 was used for concentrations of 0.23-3.50 mg NO_3^--N/L and TNT 836 for concentrations of 5-35 mg NO_3^--N/L. In some cases, three samples were taken to compare and guarantee the accuracy of the results.

3.4.6.3 Nitrite-nitrogen

Nitrite is one of the by-products of the nitrification process if adequate oxygen is not supplied. Nitrite is converted to nitrate if more oxygen is provided to the reactor. Because the first phase of the study was done on an aerobic biological setup, nitrite concentration was negligible. The second phase was conducted under aerobic/anoxic conditions, where recording nitrite concentration became more important.

In phase I, the nitrite measurements were conducted by using the IC. For the same reason as ammonia-nitrogen, unfortunately over time results of the IC became unreliable due to technical issues, and TNT Hach tests were the only method of measurement used. In the second phase, TNT Hach vials were used to measure nitrite-nitrogen concentrations; TNT 839 for 0.015–0.60 mg NO_2^--N/L and TNT 840 for 0.6–6.0 mg NO_2^--N/L. In some cases, three samples were taken to compare and guarantee the accuracy of the results.

3.4.6.4 Total nitrogen

Total nitrogen (TN) represents the total amount of nitrogen compounds in the sample, including organic and inorganic nitrogen compounds. Therefore, occasionally at the beginning of the study, TN measurements were conducted to provide better observation of the system. The main purpose of TN analysis was to regulate operational conditions, adjust aeration, and track nitrogen compounds in the effluent. When the system became stable, daily measurements were no longer necessary. The analyses were done by using TNT Hach kits; TNT 826 was used for 1–16 mg TN/L.

3.4.6.5 Total Kjeldahl nitrogen (TKN)

TKN measurements were done in order to assess the amount of organic and inorganic nitrogen compounds in the effluent. In the adaptation stage of the first phase, some unpredicted results required interpretation. Therefore, TKN tests were conducted to clarify the nitrogen composition of the system. The analysis was done by using TNT Hach kits; TNT 880, s-TKN was used for concentrations of 0–16 mg TKN/L.

3.4.7 Phosphorus concentration

In this study, phosphorus concentrations were measured on a weekly basis in order to assess phosphorus removal efficiencies. In the first phase, both the IC machine and TNT Hach kits were used to measure the phosphorus concentrations; TNT 844, Phosphorus Reactive was used for concentrations of 1.4–15 mg PO_4^{3-}/L.

In the second phase of the study, only the TNT Hach method was applied. Because the source of phosphorus in the synthetic wastewater was a soluble form of phosphorus, only reactive phosphorus was measured. When phosphorus concentrations were out of the detection range of the kits, sample dilution was necessary. In some cases, three samples were taken to compare and guarantee the accuracy of the results.

Previous studies have confirmed that SEMBR systems have a high capacity for phosphorus removal (Bani-Melhem et al., 2009). Unlike nitrogen removal, phosphorus removal is relatively stable in similar operational conditions. Therefore, measuring of phosphorus concentrations was not required on a daily basis.

Similar to the nitrogen removal analysis, the phosphorus removal was evaluated by comparing the EIFAS and the IFAS reactors efficiency. The comparative analysis between phase I and phase II was conducted to show the efficiency of the EIFAS reactor for both nitrogen and phosphorus removal.

Chapter IV

Results and Discussion

4.1 Introduction

Successful results of the two phases of the experimental study have been recorded for sixteen months. The first phase was started in June 2019 and ended in February 2020. The second phase was operated from February 2020 to September 2020 (during COVID-19 lockdown). During this period, hundreds of samples were taken and data was analysed. Evaluated data included the results of current density, DO, ORP, COD, nutrient removal, TSS, and nitrate concentrations.

Furthermore, data related to the nutrient removal, COD and nitrate concentrations in two phases were compared. Subsequently, the trends of nitrate and nitrite generation were then compared with ammonia removals. In addition, the nutrient and COD removal capacity of attached cultures to media in are shown in section 4.5. In this study, variable parameters were aeration and ammonia inflow. A part of the experiment results was influenced by controlling parameters and the adaptation period. Furthermore, suggested ranges of DO and current density were provided the optimal nutrient removal results.

4.2 Dissolved oxygen (DO) concentration, phase I

Control of dissolved oxygen is one of the most important factors used in wastewater treatment; aeration rate directly affects the nitrification/denitrification process. Therefore, adequate aeration was continuously provided during the first phase of the study. In the second phase, aeration supply was carefully controlled in order to balance the amount of oxygen required for coexistence of nitrifiers and denitrifiers. Thus, required aeration rate in phase II was much lower than in phase I. Figure 4-1 shows the result of dissolved oxygen concentrations (mg/L) in phase I, which lasted from the 25th of June 2019 to February 3rd of 2020.

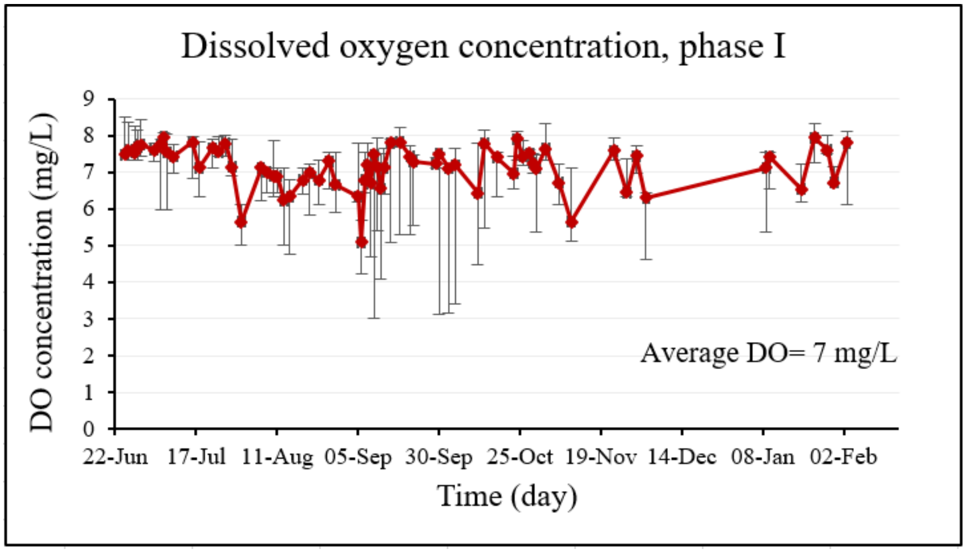


Figure 4-1 DO concentration (mg/L) in phase I

Provided aeration created a condition, which would helped nitrifying bacteria grow faster and oxidize ammonia in phase I. DO was regularly supplied to the reactor in a range of 6-8 mg/L, unless an unexpected mishap occurred. DO concentration curve fluctuations were mainly caused by adjustments of the aeration supply during the experiment. These adjustments were conducted based on removal results, bacteria biomass color and air bubbles observed.

The concentration of dissolved oxygen in phase II changed only marginally with switching OFF the current input. The dissolved oxygen level continuously decreased a few seconds after the current input was turned ON. When current input was turned OFF, the dissolved oxygen began to increase within seconds. These changes recovered when the mode was switched, and the reactor recovered its previous condition.

Since the reactor was relatively small compared to pilot scale systems, air distribution was more of a challenge. Dissolved oxygen levels are more easily maintained and controlled in larger-scale operations.

4.3 Oxidation-reduction potential, phase II

ORP measurements were conducted in this experiment in order to assess the sludge condition; ORP values used for this purpose are shown in figure 4-2 and were recorded during the OFF mode. ORP values were also recorded during the ON mode; these values were used to show the effect of the electrical field in ON and OFF states. A clear ORP value distinction between the ON and OFF modes has been observed.

Considering that the DO probe was not available in the second phase, measurement of ORP values was used to estimate the level of oxidation/reduction in the reactor. Changes in ORP were related to the current input in the EIFAS reactor under similar aeration input. Subsequently, the current changed dissolved oxygen uptake of microorganisms as well as chemical reactions through which oxygen was consumed. Oxic/anoxic conditions followed OFF/ON intermitted current creating adequate conditions for nitrifiers/denitrifiers, respectively. The random ORP measurements permitted to assess the range of ORP fluctuation. Figure 4-2 shows these measurements of the oxidation-reduction potential in phase II.

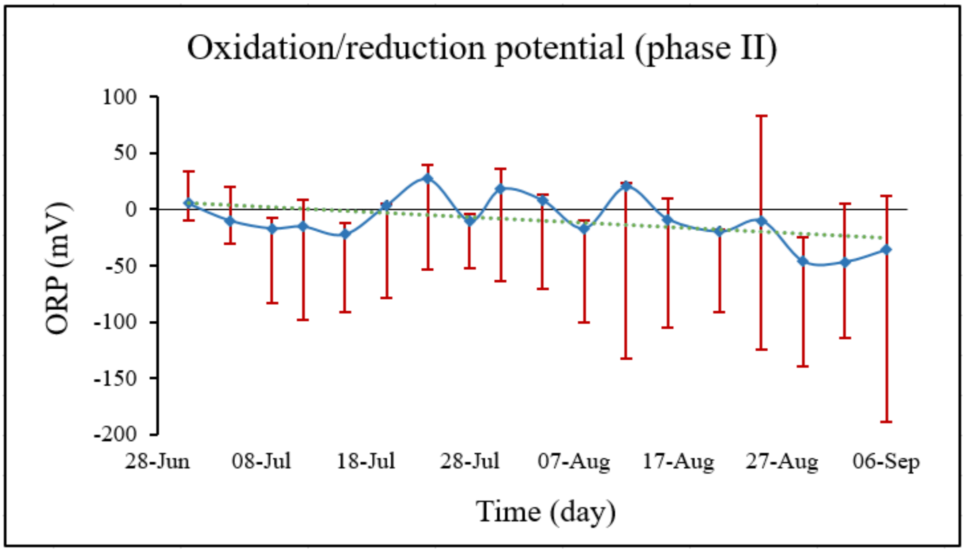


Figure 4-2 Oxidation-reduction potential (ORP) (mV) in phase II

These measurements showed that the last stage of this study was done under the highest anoxic conditions, where ORP declined to -189 mV.

4.4 Nitrogen removal, phase I

Nitrogen in the wastewater is mostly in the form of ammonium. During the wastewater treatment process, nitrite and nitrate are generated as a chemical reaction by-product. Therefore, all these three forms of nitrogen compounds were measured.

According to the classification of this study shown in table 3-1, after the design and adaptation stages, the IFAS reactor was examined to find the trend, which microorganisms had in the system. Ammonium removals and generated nitrate concentrations recorded in the first and second phase of this study are presented in section 4.4 and 4.7, respectively.

4.4.1 Ammonia-nitrogen, phase I

Since ammonia has a negative impact on the environment, one of the major objectives of wastewater treatment is to eliminate it. The nitrification/denitrification process converts a majority of ammonia in to nitrate and then, in to nitrogen gas.

Ammonium sulphate was the only source of nitrogen used in the making of synthetic wastewater in this project. The C:N ratio affects nitrogen removal (Gao, 2014); a ratio between 3:1 and 1:1 was assumed for C:N. For this reason, an adequate carbon source was provided to the microorganisms to omit this effect.

The first phase began on June 20th of 2019 and ended on February 3rd of 2020, where ammonia-nitrogen was measured daily. Figure 4-3 shows the result of the ammonia-nitrogen removal concentration in the first phase of the study.

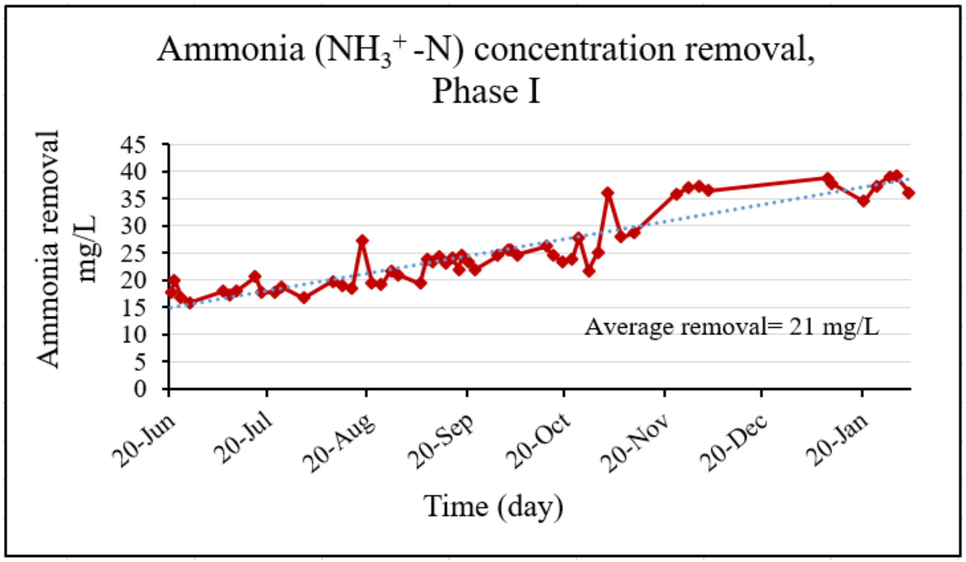


Figure 4-3 Ammonia-nitrogen removal (mg/L) vs. time in phase I

The average ammonia-nitrogen removal in this phase was 21 mg/L; it varied between 15.9 mg/L and 39.2 mg/L. The trend of the ammonia-nitrogen removal showed that the operation had improved over time. This could be due to the fact that attached biofilm and suspended culture combination performed actively to remove nutrient in the IFAS reactor.

4.4.2 Nitrate-nitrogen, phase I

Nitrate-nitrogen is another main form of nitrogen compound found in wastewater. It is the final product of the ammonia nitrification process. Nitrate converts to nitrogen gas in the final step of denitrification process. In this study, the nitrogen was supplied by adding ammonia sulphate to the synthetic wastewater. Therefore, the amount of nitrate-nitrogen measured in the effluent was mostly derived from the nitrification process in the reactor.

The first phase of the study was started on June 20th, 2019 and lasted until February 3rd, 2020. A aerobic conditions were dominant most of the experiment. This is how ammonia oxidation conditions were met and why nitrate reduction was not taking place as intended. The effluent after microfiltration was tested to find the nitrate-nitrogen concentration. Since nitrate was not added to the influent, nitrate in the effluent was produced due to the ammonia oxidation process. Figure 4-4 shows the effluent nitrate-nitrogen concentration in first phase.

The average nitrate-nitrogen concentration in the first phase was 16 mg/L while the concentration varied between 0.495 mg/L and 31 mg/L. When the ammonium removal increased over time and that nitrate was generated because of ammonium oxidation, the effluent nitrate concentration increased. Several fluctuations in the nitrate-nitrogen concentration were observed, which were assumed to be a result of the operation conditions, improper mixing, and fluctuation of the influent ammonia concentrations.

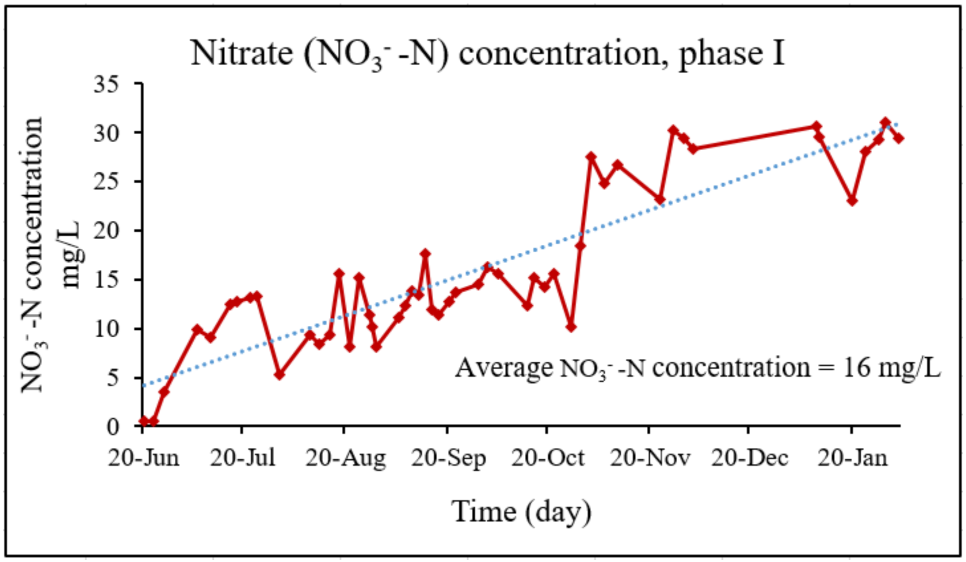


Figure 4-4 Effluent nitrate-nitrogen concentration (mg/L) in phase I

4.4.3 Ammonia-nitrogen versus nitrate-nitrogen in IFAS (phase I)

The concentration changes in ammonia-nitrogen and nitrate-nitrogen indicate whether a sludge is in the process of nitrification or denitrification. An increase of nitrate-nitrogen and a decrease of ammonia-nitrogen in the effluent indicated the level of nitrification in the reactor under oxidation conditions.

The amount of ammonia-nitrogen removal increased during the ten months of study and finally reached 39 mg/L and nitrate-nitrogen concentration increased to 31 mg/L. The aerobic condition in the first phase provided a suitable environment for nitrifying bacteria where ammonia was oxidized in the nitrification process. This condition was not desirable for denitrifying bacteria to grow and actively participate in the denitrification process. Therefore, nitrate concentration increased in the reactor. The ammonia-nitrogen removal and nitrate-nitrogen concentration in phase I is presented in figure 4-5.

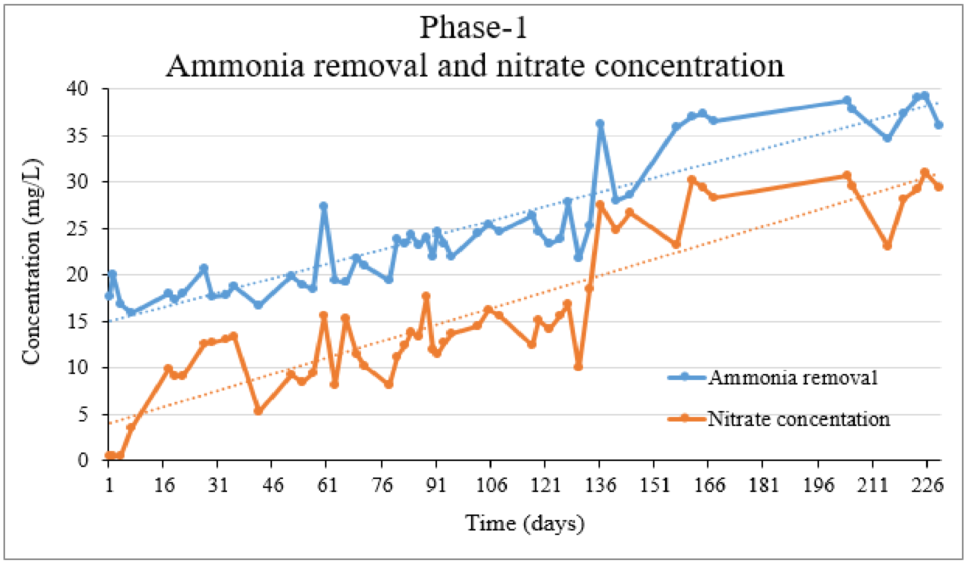


Figure 4-5 Ammonia-nitrogen concentration removal and nitrate-nitrogen concentrations (mg/L) in phase I

4.5 Efficiency trials of the attached and suspended cultures (phase I, stage 4)

The IFAS reactor is a combination of the fixed-film covering the surface of media and suspended sludge in a reactor. This combination raises a question about the contribution of each of these two cultures in overall nutrient removal and nitrate production. Therefore, an experimental evaluation was designed to measure the effect and involvement of the attached biofilm or suspended biomass.

The reactor was separated in to two compartments (suspended biomass and media) in order to conduct this comparative study. The media was later transferred to second reactor with tap water as its liquor. In another reactor, suspended biomass was in a conventional return activated sludge process. The media reactor was under similar conditions to the activated sludge reactor in terms of DO, HRT, and influent nutrient concentrations.

This test was conducted six times during phase I of this study; after 6, 10, 13, 15, 21, and 29 weeks. The results are presented from section 4.5.1 to 4.5.6. Each trial lasted eight days which ensured at least four stable readings would be recorded.

4.5.1 Six weeks after phase I began

Six weeks after starting phase I, where media and suspended biomass were combined in the IFAS reactor, the first analyses have been conducted. On the first day of trials, effluent of the media reactor did not contain any nutrient including ammonia and nitrate. Effluent and initial liquor in the media reactor were water in the first day, an apparent high removal efficiency was recorded. After four days of trials, stable results were observed, which were not influenced by the initial liquor in the media reactor. The results of day 4,5,6,7 and 8 were recorded. Therefore, the average of results recorded from day 5 to day 8 was considered as the removal efficiency of the media reactor.

In the activated sludge reactor, the first day's ammonia removal and nitrate concentration were measured immediately after separation of media from the combined IFAS reactor. Effluent supernatant taken from the activated sludge reactor was influenced by fixed-film on the surface of media. Real ammonia removal results for activated sludge began to emerge after 4 days. The average results between day 5 and day 8 had been considered as a treatment efficiency of the activated sludge reactor.

Ammonia-nitrogen removals and nitrate-nitrogen concentrations in the effluent in both comparative reactors six weeks after phase I began are shown in figures 4-6 and 4-7 respectively. The average DO concentrations in the media reactor and in the AS reactor were 6.05 mg/L and 6.78 mg/L respectively. The sum of the ammonia removal results and total nitrate concentrations are presented in each graph. Involvement and efficiency improvement of the attached biofilm and suspended biomass were found by comparing the removal results from each and combined reactor as well as the total removal by AS and media reactors.

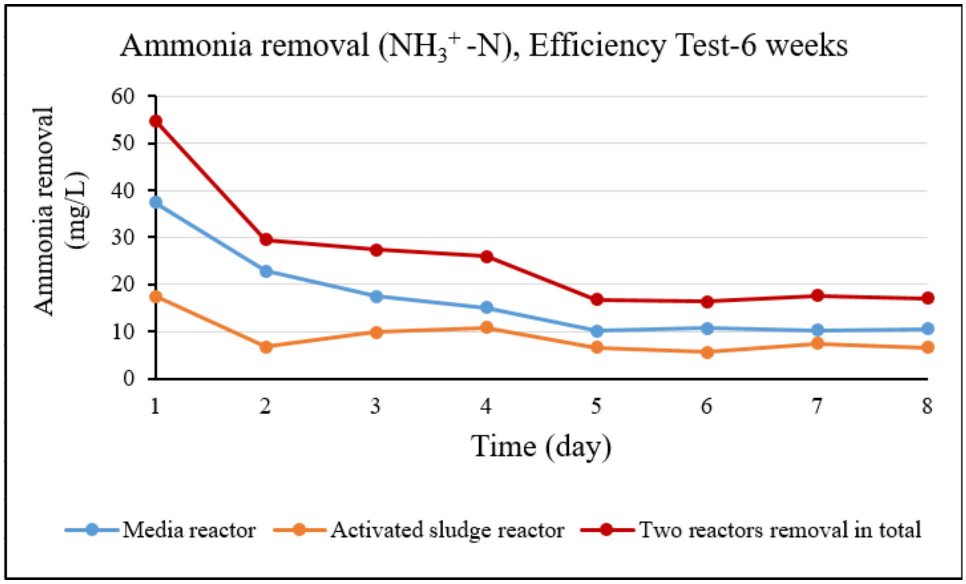


Figure 4-5 Amount of ammonia-nitrogen removed in both reactors six weeks after phase I began

The average ammonia-nitrogen removal reported from July 1st to August 1st (This period ended before the first set of comparative studies conducted), was 18 mg/L. The ammonia removal recorded on August 1st was 17.4 mg/L, which was the same as the reported concentration removal value in the activated sludge reactor on the first day. The average ammonia-nitrogen concentration removal for the last four days of the test was 6.55 mg/L in the activated sludge reactor and 10.3 mg/L in the media reactor. The sum of ammonia-nitrogen concentration removal for both reactors was 15.85 mg/L. This result was similar to the average ammonia concentration removed from the IFAS reactor, a month before efficiency analyses were done.

The nitrate-nitrogen concentration is another important parameter used to evaluate the role of fixed-film on media and suspended biomass in the IFAS reactor's performance.

The average nitrate-nitrogen concentration reported for IFAS from July 1st to August 1st was 10.8 mg/L. Moreover, the nitrate-nitrogen concentration on August 1st was 8.35 mg/L in the IFAS reactor, which was the same as the activated sludge reactor concentration on day one.

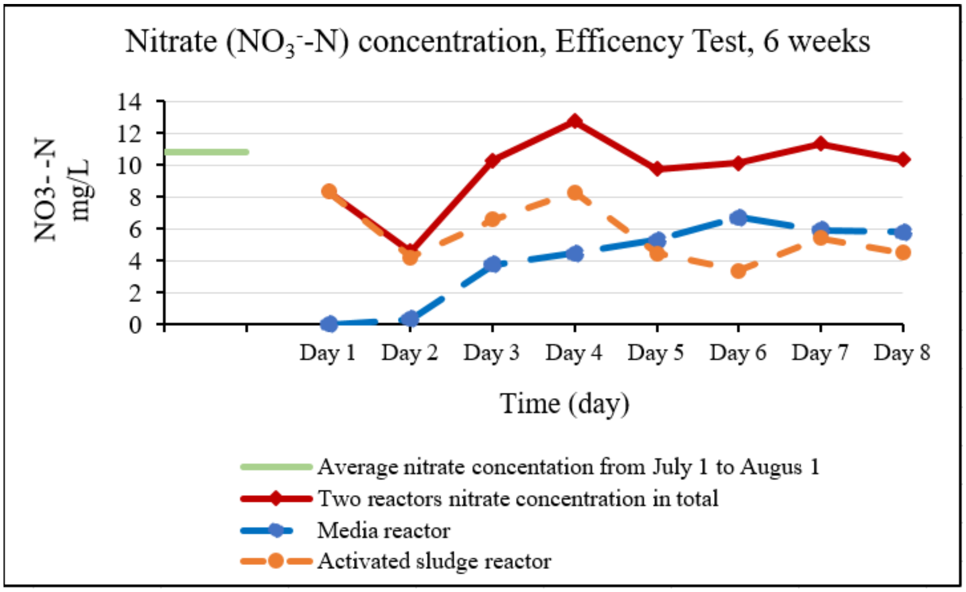


Figure 4-6 Nitrate-nitrogen concentration in both reactors six weeks after phase I began

The average nitrate-nitrogen concentration for the last four days of the test was recorded 4.44 mg/L in the activated sludge reactor and 5.93 mg/L in the media reactor. The average sum of nitrate-nitrogen concentration for two reactors was 10.3 mg/L, which It matched the average nitrate-nitrogen concentration in the IFAS reactor after a month of testing.

A linear relation between the sum of nitrate-nitrogen concentrations in the two reactors and IFAS reactor was observed. In the first batch of tests done to assess fixed-film and suspended biomass efficiency. However, the results of ammonia-nitrogen concentration removal were less than that of the IFAS reactor. Moreover, the amount of ammonia-nitrogen removed and the nitrate concentration in the media reactor were higher than the values recorded in the activated sludge reactor. About 61% of the ammonia-nitrogen removed from the two reactors was a result of nitrification in the media reactor, versus 39% in the AS reactor. Furthermore, the nitrate-nitrogen concentration in the media reactor was 57% of the sum of the nitrate-nitrogen concentrations recorded in two separated reactors.

4.5.2 Ten weeks after phase I began

Stable results (in steady-state conditions) are fundamental for the comparison of the efficiency of these two reactors; the fixed-film reactor and activated sludge reactor. In batch tests, ammonia removal and nitrate concentration results reached the stable performance on day 3 and day 4 respectively. The averages DO concentration in the media reactor and activated sludge reactor were 7.96 mg/L and 7.88 mg/L, respectively.

Average ammonia-nitrogen removal in the media reactor and activated sludge reactor were 12.1 mg/L and 7.1 mg/L, respectively. Thus, an overall ammonia-nitrogen removal of 19.2 mg/L was obtained from these two reactors. The media reactor removed 63% of overall ammonia-nitrogen. It means that the attached culture effectively participated in the nitrification process. Moreover, the contribution of the media reactor in the total ammonia-nitrogen removal of two reactors increased from 61% for the batch test after six weeks to 63% for the batch test after 10 weeks.

The average ammonia-nitrogen removal was recorded as 20.7 mg/L from August 9th to August 29th in the IFAS reactor. The efficiency results of the IFAS reactor and the sum of efficiency of these two reactors (media reactor and activated sludge reactor) were close.

Figures 4-8 and 4-9 show the ammonia-nitrogen concentration removal and the effluent nitrate-nitrogen concentrations, in comparative reactors ten weeks after phase I began.

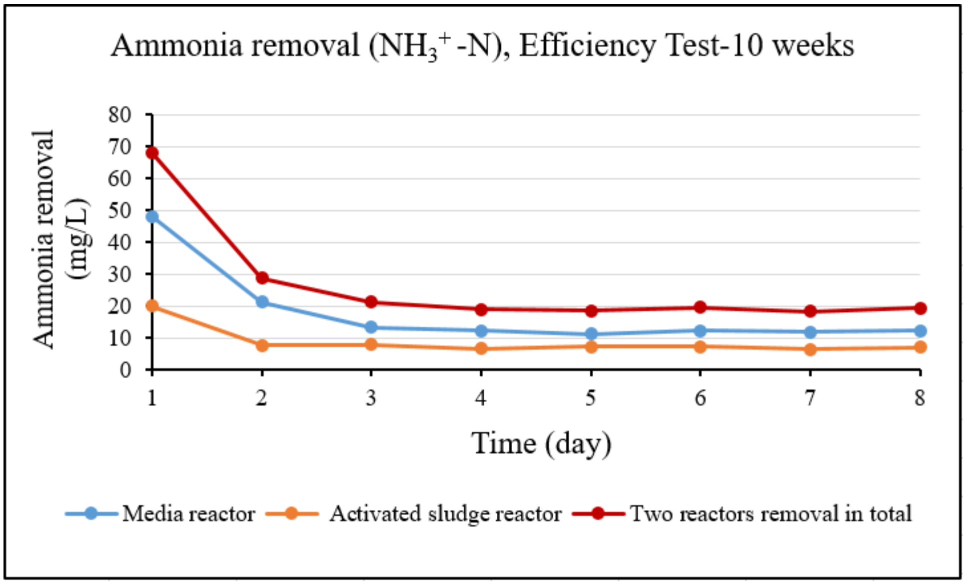


Figure 4-7 Amount of ammonia-nitrogen removed in both reactors ten weeks after phase I began

The average effluent nitrate-nitrogen concentration in media reactor and activated sludge reactor were 8.26 mg/L and 4 mg/L, respectively. A nitrate-nitrogen concentration of 12.2 mg/L was estimated in the combined IFAS reactor; however, nitrate concentration was found to be 10.6 mg/L in the IFAS. The reason behind it may be that excessive aeration in two reactors, which did not allow the same level of denitrification process to take place in each reactor. Therefore, the sum of nitrate-nitrogen concentrations was higher than the concentration in IFAS reactor.

About 67% of the nitrate build-up in the two rectors was generated in the media reactor, and the remaining is a result of nitrification in the activated sludge reactor.

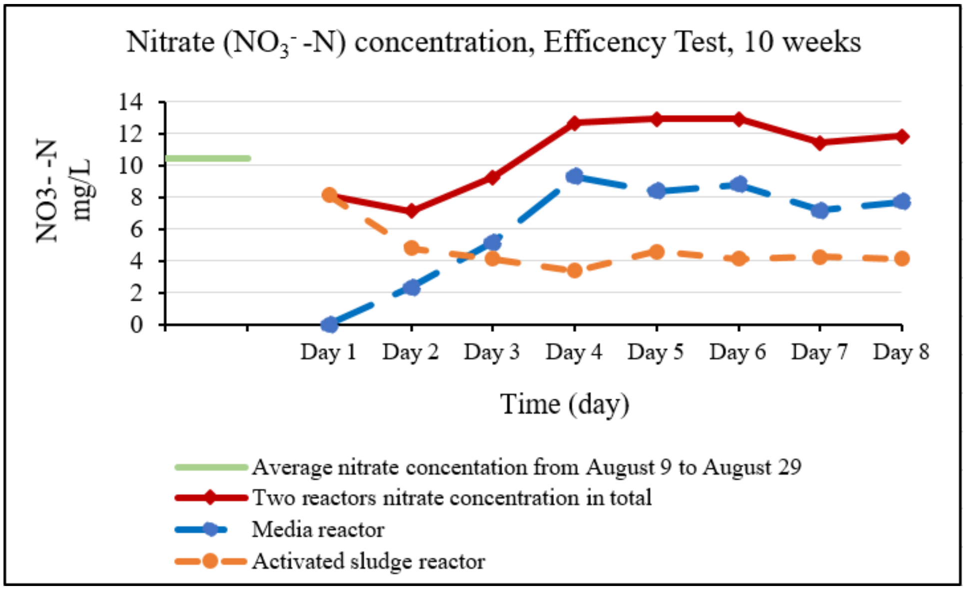


Figure 4-8 Nitrate-nitrogen concentration in both reactors, ten weeks after phase I begun

4.5.3 Thirteen weeks after phase I began

The averages of DO concentration in the media reactor and the activated sludge reactor were 6.06 mg/L and 5.56 mg/L for eight days of this batch of tests. The average ammonia-nitrogen removal in media reactor was 17.8 mg/L and in activated sludge was 19.8 mg/L. In total, two reactors removal was 37.6 mg/L, together. Nevertheless, the average removal of the combined reactor (IFAS) between 5th of September and 22nd of September was 23 mg/L.

Unexpected results were verified by another batch of test two weeks after this batch. Figure 4-10 shows the ammonia-nitrogen removal for two reactors thirteen weeks after phase I began.

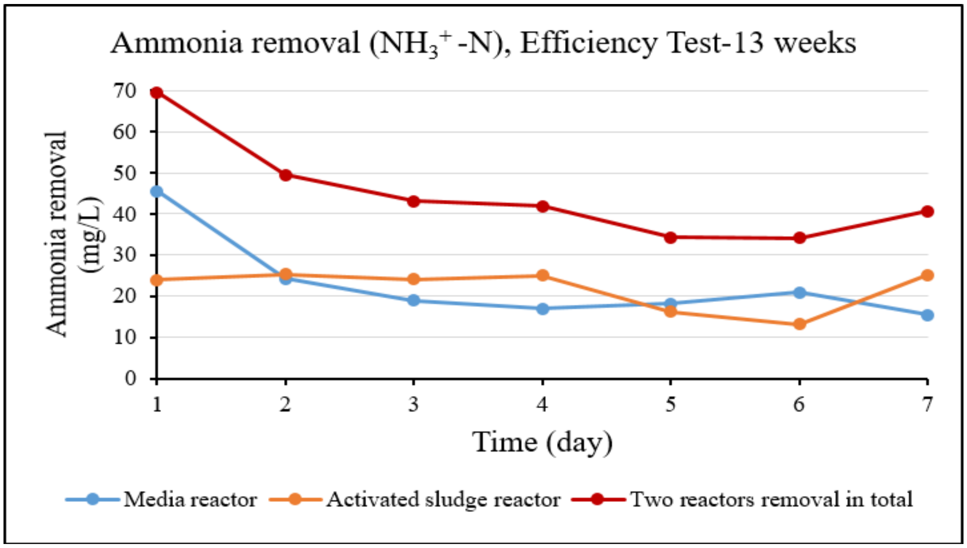


Figure 4-9 Amount of ammonia-nitrogen removed in both reactors 13 weeks after phase I began

Both ammonia removal and nitrate concentration reached stability after day 2. The average (from day 2 to day 7) nitrate-nitrogen concentration in media reactor and activated sludge reactor were 3.12 mg/L and 8.65 mg/L respectively. The average effluent nitrate-nitrogen concentration in the IFAS reactor was recorded 13 mg/L; from September 5th to September 22nd. Moreover, the sum of nitrate-nitrogen concentration in two reactors of efficiency test was 11.8 mg/L. It means that denitrification was stronger in these two reactors than in the combined reactor.

About 53% of the total ammonia removal of two reactors took place in the AS reactor, versus 47% in the media reactor. About 26% of the nitrate build-up in the two rectors was generated in the media reactor, and the 74% was a result of nitrification in the activated sludge reactor.

Therefore, this result shows that the micro biofilm created adequate conditions for denitrification in the media reactor; show better results than activated sludge reactor. Figure 4-11 shows comparative results for the nitrate-nitrogen concentration in two reactors thirteen weeks after the IFAS reactor was operated.

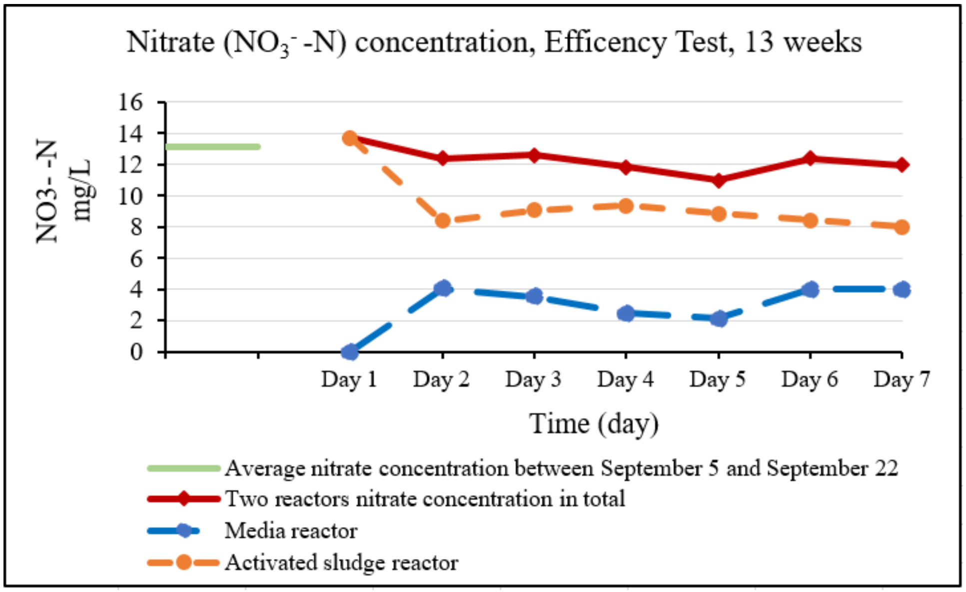


Figure 4-10 Nitrate-nitrogen concentration in both reactors 13 weeks after phase I began

4.5.4 Fifteen weeks after phase I began

The unexpected results of ammonia-nitrogen removal led to conduct another batch of efficiency tests to evaluate a contribution of media fixed-film and suspended biomass to the final efficiency of the combined reactor. Figure 4-12 shows the results of ammonia-nitrogen concentration removal and the total concentration removal of both reactors.

The averages of DO concentration in the media reactor and the activated sludge reactor were 5.56 mg/L and 5.06 mg/L for eight days of this batch of tests. The average ammonia-nitrogen removal in media reactor was 15.4 mg/L and in activated sludge was 10.75 mg/L. In total, both reactors removed 26.15 mg/L. Around 49% of the ammonia removal took place in the activated sludge reactor versus 51% in the media reactor.

Moreover, the average ammonia removal of 25.1 mg/L was recorded in the IFAS reactor from October 1st to October 5th. This value was close to ammonia removal in media reactor plus in AS reactor, which was 26.15 mg/L. Figure 4-12 shows the comparative ammonia-nitrogen removal results fifteen weeks after phase I began.

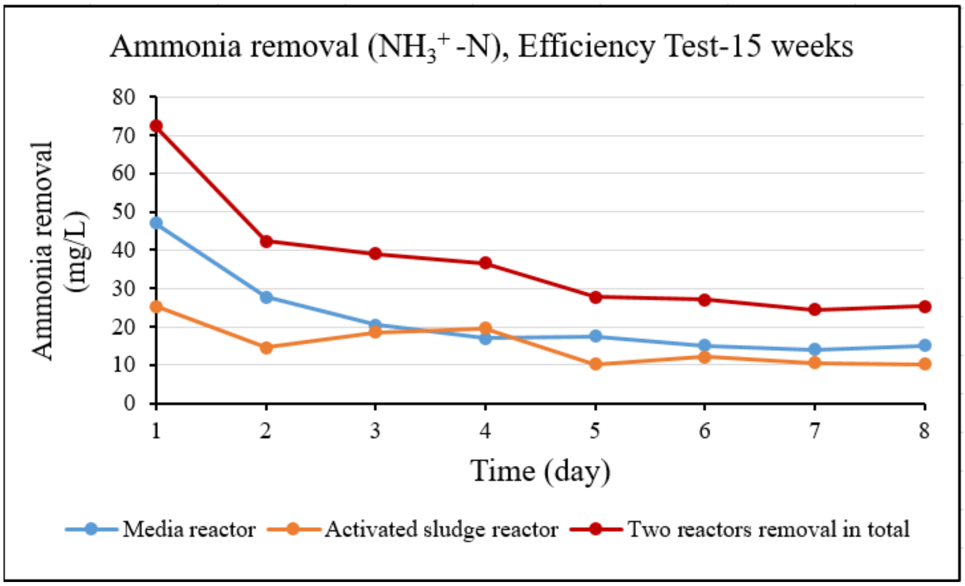


Figure 4-11 Amount of ammonia-nitrogen removed in both reactors 15 weeks after phase I began

The nitrate concentration became stable after 4 days. The average nitrate-nitrogen concentration for the last four days of the test was recorded 6.5 mg/L in the activated sludge reactor and 8.52 mg/L in the media reactor. The sum of these two concentration values are15.02 mg/L. About 57% of the nitrate build-up in the two rectors was generated in the media reactor, and the 43% was a result of nitrification in the activated sludge reactor.

Moreover, average nitrate-nitrogen concentration in effluent was recorded 15.4 mg/L in the IFAS reactor between October 1st and October 5th; it shows accordance between the values. Figure 4-13 shows the results of 8-days comparative batch tests in two comparative reactors with respect to the nitrate-nitrogen concentration fifteen weeks after phase I began.

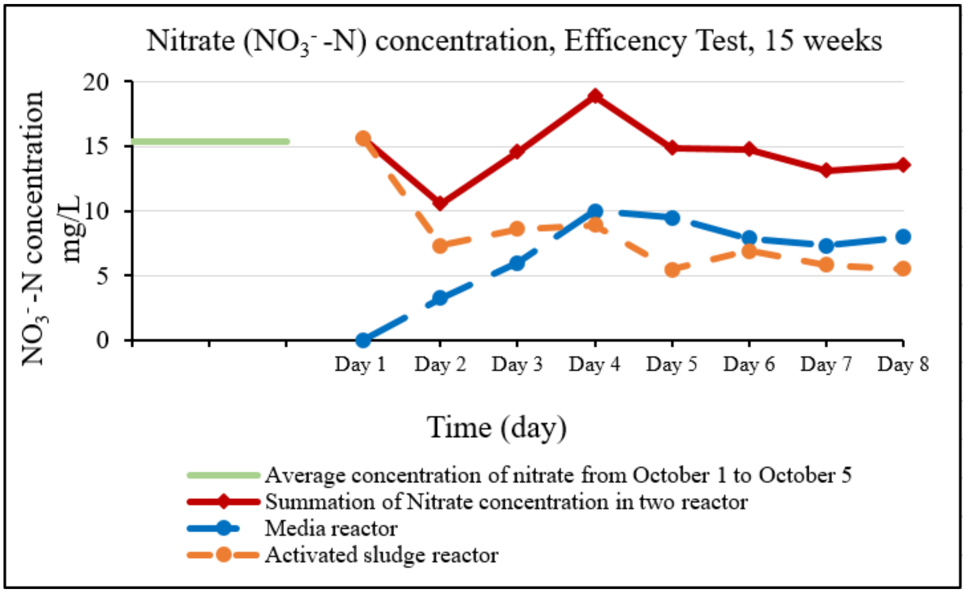


Figure 4-12 Nitrate-nitrogen concentration in both reactors 15 weeks after phase I began

4.5.5 Twenty-one weeks after phase I began

The averages DO concentration in the media reactor and activated sludge reactor were 6.1 mg/L and 5.56 mg/L, respectively. The ammonia removal results became stable after two days.

Average ammonia-nitrogen removal in the media reactor and activated sludge reactor were 15.3 mg/L and 11.7 mg/L, respectively. Thus, an overall ammonia-nitrogen removal of 27 mg/L was obtained from these two reactors. In the media reactor, 56% of ammonia removal took place versus 44% in the activated sludge reactor.

Moreover, the average removal of the combined IFAS reactor between 14th October and 12th November was 26.6 mg/L, which is close to sum of removal from two separated reactors (27 mg/L). Figure 4-14 shows the ammonia-nitrogen removal for two comparative reactors twenty-one weeks after phase I began.

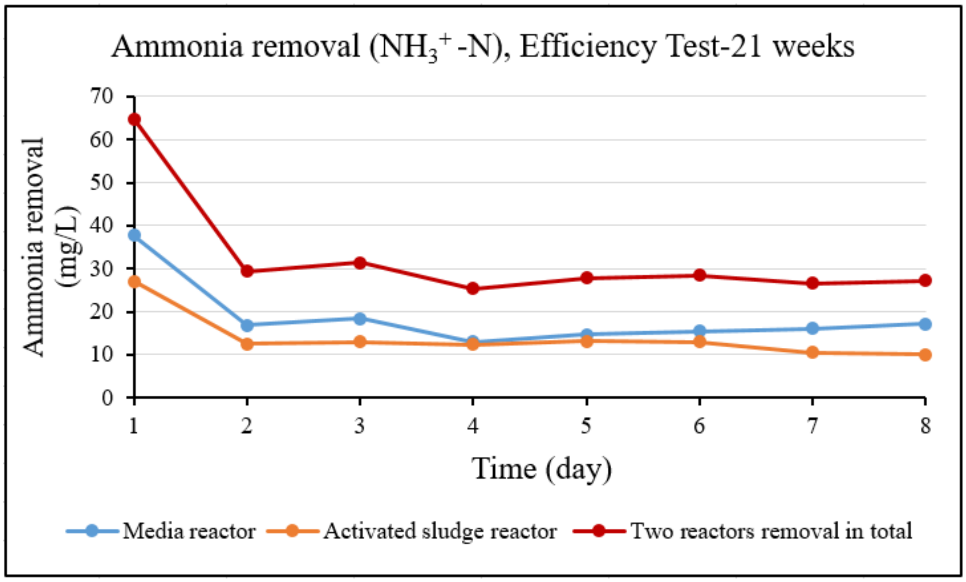


Figure 4-13 Amount of ammonia-nitrogen removed in both reactors 21 weeks after phase I began

The average effluent nitrate-nitrogen concentration in media reactor and activated sludge reactor were 12.9 mg/L and 10.4 mg/L, respectively. A nitrate-nitrogen concentration of 23.3 mg/L was estimated in the combined IFAS reactor; which is close to average recorded values of 24.3 mg/L of nitrate concentration in the IFAS from October 30th to November 12th. The accordance of these two values shows that the efficiency of combined IFAS reactor was superposition of the media reactor and suspended sludge reactor efficiencies.

About 55% of the nitrate build-up in the two rectors was generated in the media reactor, and the 45% was a result of nitrification in the activated sludge reactor. Figure 4-15 shows the comparative nitrate-nitrogen concentration and the sum of concentration in two reactors twenty-one weeks after phase I began.

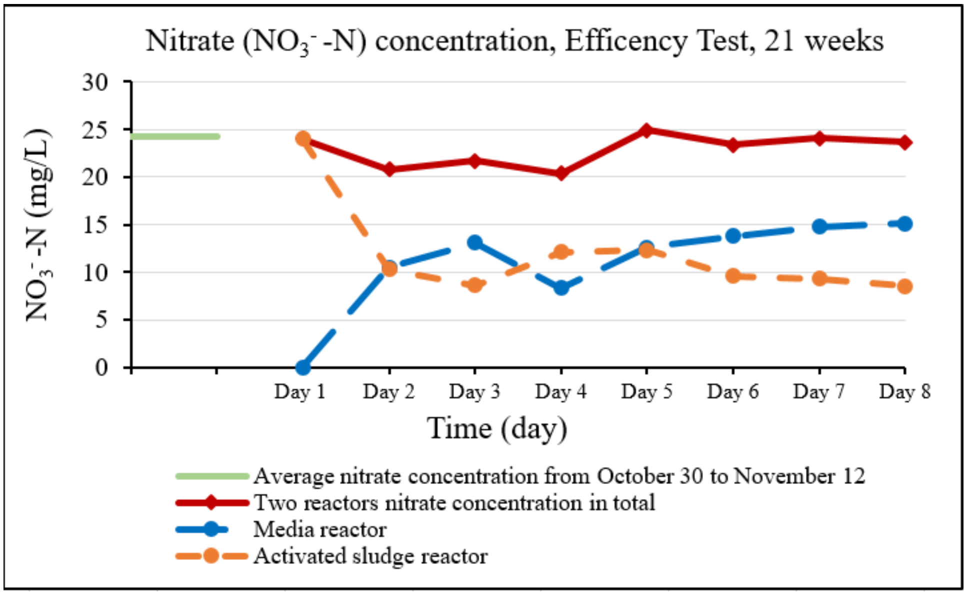


Figure 4-14 Nitrate-nitrogen concentration in both reactors 21 weeks after phase I began

4.5.6 Twenty-nine weeks after phase I began

Twenty-nine weeks after starting point, the 8-day batch tests showed an average DO concentrations of 7.2 mg/L and 7.5 mg/L in media and activated sludge reactors, respectively. Figure 4-16 shows results of the ammonia-nitrogen concentration removal in compared reactors after twenty-nine weeks from the starting point of the IFAS reactor.

These DO levels show that the sludge was under aerobic conditions entire time. Theoretically, the nitrification reaction will be stronger than denitrification when DO concentration is high. An increased ammonia removal and more of nitrate-nitrogen production proved this assumption.

The average ammonia-nitrogen removal in the media reactor was 23.5 mg/L and in the activated sludge reactor was 13.7 mg/L, which correspond to 63% and 37% removal, respectively. Overall, 37.2 mg/L of ammonia-nitrogen was removed in both reactors. The significant results of this batch test were equal to the average ammonia-nitrogen removal in the IFAS reactor from November 20th to 10th January (37.2 mg/L).

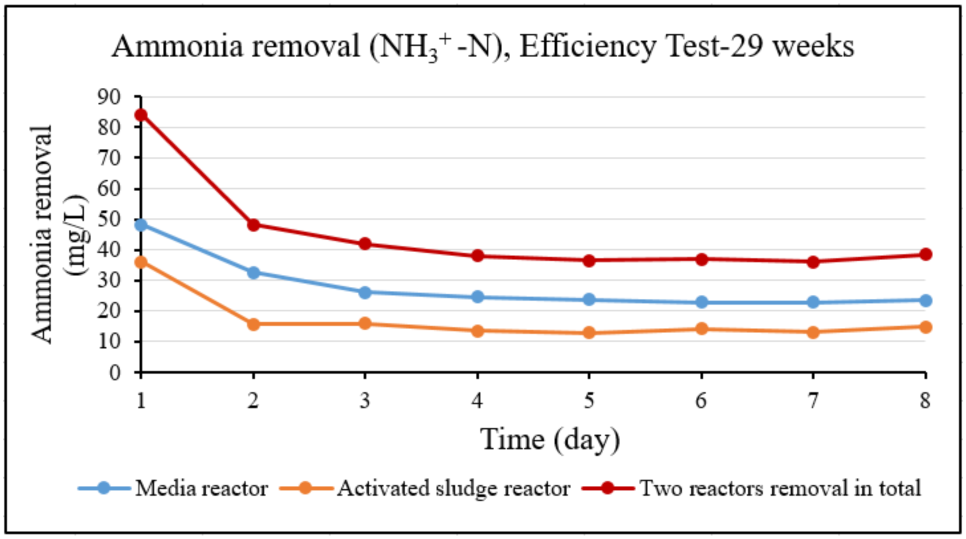


Figure 4-15 Amount of ammonia-nitrogen removed in both reactors 29 weeks after phase I began

Another important parameter, which evaluates the efficiency of fixed-film and suspended biomass, is the generated nitrate concentrations. Figure 4-17 shows the results of nitrate-nitrogen concentration in the two reactors after twenty-nine weeks from starting point of the IFAS reactor.

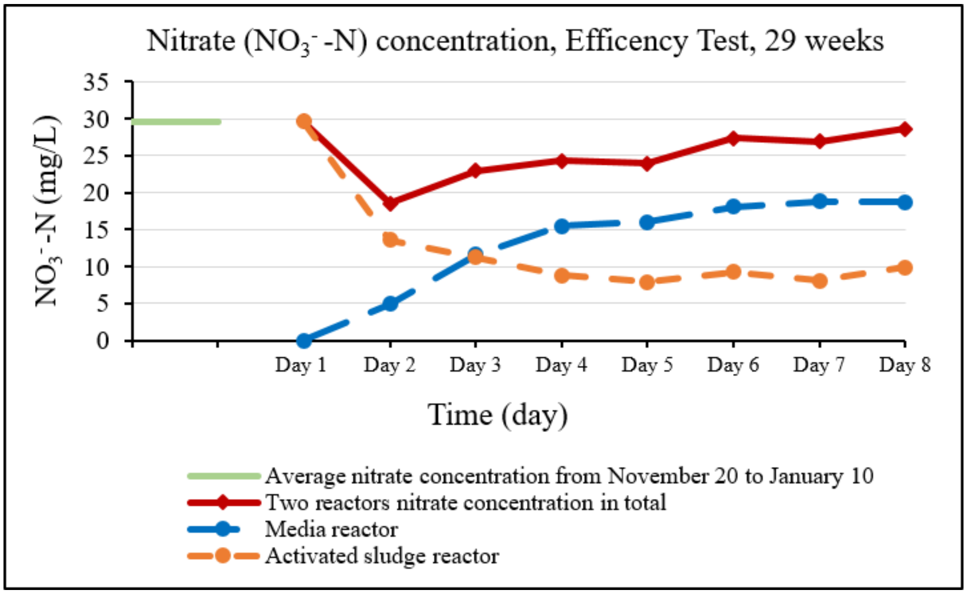


Figure 4-16 Nitrate-nitrogen concentration in both reactors 29 weeks after phase I began

The average nitrate-nitrogen concentration in the media reactor was 17.4 mg/L and in the activated sludge reactor was 8.8 mg/L, which respectively correspond to 67% and 33% of total concentration value. Total nitrate-nitrogen build-up in two reactors were 26.2 mg/L, whereas 29.6 mg/L of nitrate-nitrogen concentration in the IFAS reactor. It indicated that the denitrifying bacteria were more active in the separate reactors than in the combined IFAS reactor.

4.5.7 Summary of comparative study of attached culture and suspended biomass

In order to understand an impact of the attached and suspended culture comparative study was conducted in phase I. This eight days study was repeated five times; at 6, 10, 15, 21, and 29 weeks after phase I was started. Results reached stability after four days in each set of the comparative studies. To assess the trend of results for the comparative study, stable results of each set are discussed in this section. Figure 4-18 shows the trend of ammonia removal in comparative studies done in the first phase.

Ammonia removal in the media reactor improved from 10 mg/L after 6 weeks to 23.7 mg/L after 29 weeks. During the same period, the ammonia removal in the activated sludge reactor changed from 6 mg/L to 14 mg/L. Nitrification done by biofilm was stronger than by suspended culture, since nitrifying bacteria were attached to the media. Moreover, these studies proved that ammonia removal in the IFAS reactor was superposition of ammonia removal in the media biofilm and activated sludge reactors.

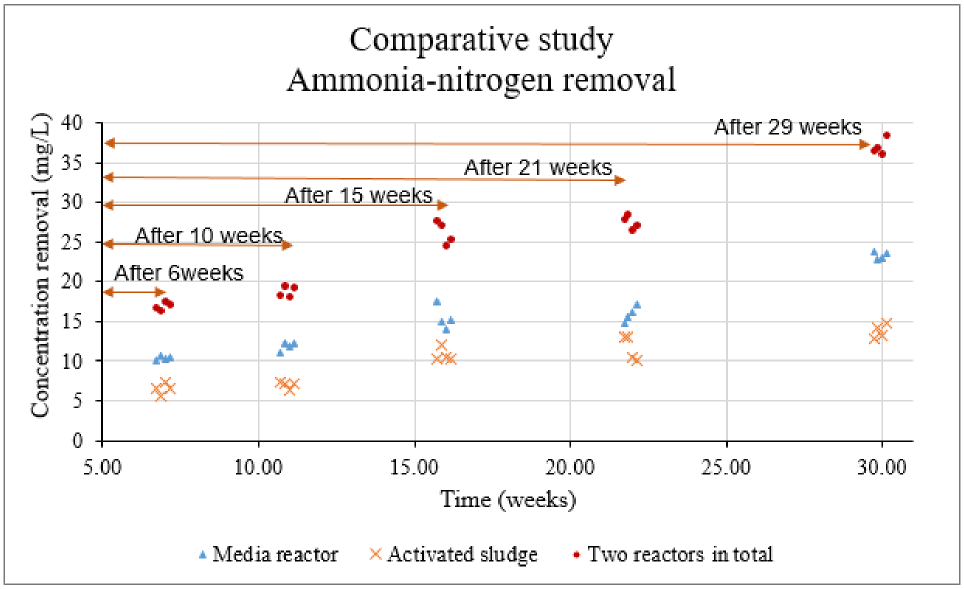


Figure 4-18 Trend of ammonia-nitrogen removal in comparative studies done over 29 weeks

Nitrate is generated in the reactor in the ammonia oxidation process. Considering that ammonia oxidation increased overtime, the nitrate concentration in each set of the tests increased in comparison to previous sets. The trend of nitrate-nitrogen concentration in the activated sludge reactor, media reactor, and the sum of the two reactors is presented in figure 4-19.

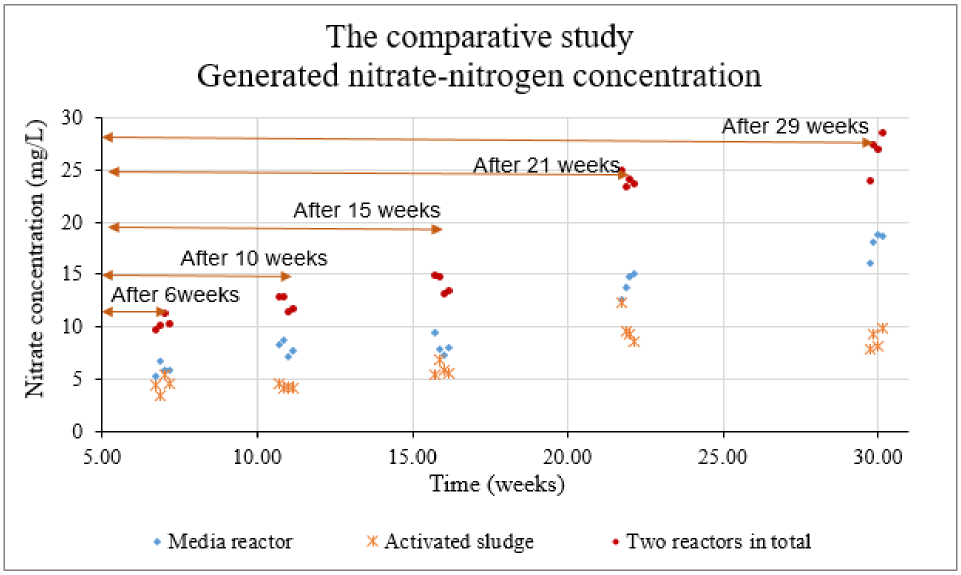


Figure 4-19 Trend of nitrate-nitrogen concentration in comparative studies done over 29 weeks

The nitrate-nitrogen concentration in the media reactor increased to 18 mg/L versus 10 mg/L in the activated sludge reactor 29 weeks after phase I was initiated. Similar to the ammonia removal, nitrate concentration in the IFAS reactor was superposition of nitrate concentration in the media reactor and activated sludge reactor. Superposition happened because nitrate removal capacity of the system in the denitrification process comes from the capacity of attached fixed-film to media and suspended biomass in sludge.

Unlike ammonia removal, carbon removal results were better in the activated sludge reactor when compared to the media reactor. The COD removal increased in the media reactor over time in each set of comparative tests done, while it would increase in the activated sludge reactor. Carbon oxidizing bacteria are fast growing, and therefore the activated sludge reactor contained plenty of carbon oxidizing bacteria in aerobic conditions. On the other hand, culture attached to media was mostly nitrifiers, not carbon oxidizing bacteria. The trend and results of COD removal in comparative studies are presented in figure 4-20.

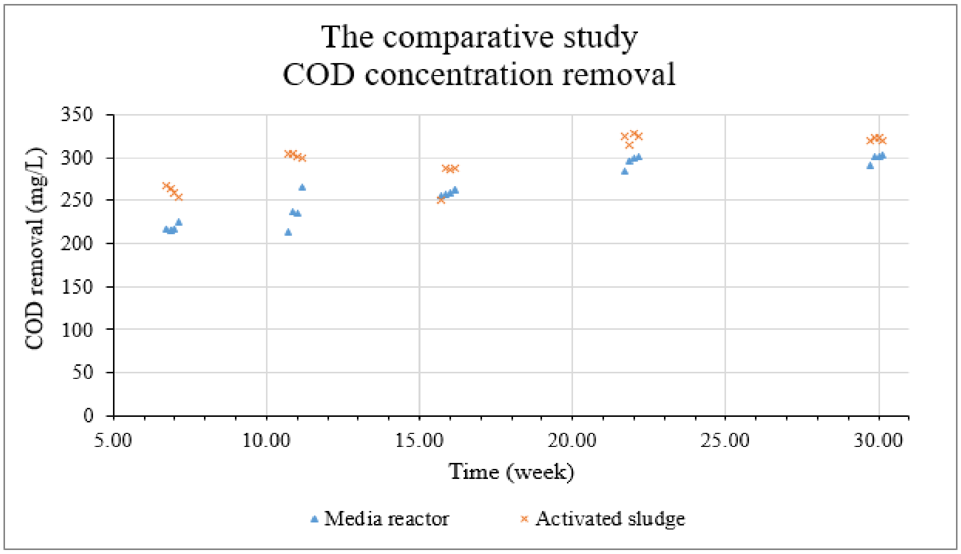


Figure 4-20 Trend of COD removal in comparative studies done over 29 weeks

4.6 Oxygen uptake rate, phase I

To evaluate growth of biofilm on the media surface over time, the oxygen uptake rate test was conducted eight times over twenty-two weeks in the first phase of the study. In this batch test, a one-liter beaker was filled with plastic media and synthetic wastewater, 30 and 70 percent by volume respectively. The mixture of wastewater and media was fully saturated with oxygen at the beginning of the test (DO concentration was around 8 mg/L). Then, the level of dissolved oxygen in the beaker was recorded every minute for a period of 20 minutes. The dissolved oxygen evolution and rate of oxygen uptake by microorganisms are shown in figure 4-21.

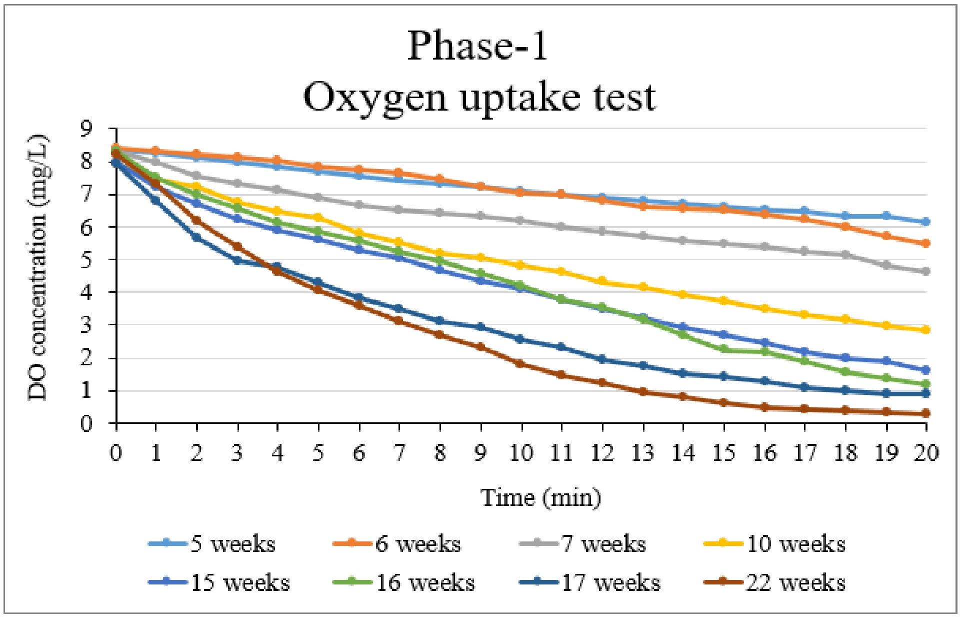


Figure 4-21 Dissolved oxygen concentration in the oxygen uptake rate test, phase I

DO concentration decreased during each twenty-minute observation, due to activity of biofilm developed on the media surface. The oxygen was up taken during bacteria growth, where nutrients present in wastewater were also used. The rate of oxygen uptake increased over time due to the fact that stronger culture was built up on the media surface.

4.7 Nitrogen removal, phase II

The second phase of the study started on 25th of February 2020 and ended on 1st of September 2020. At least twice per week, the concentration of nitrogen compounds were measured. Moreover, the ORP level was kept between in the range of aerobic and anoxic conditions.

4.7.1 Ammonia-nitrogen, phase II

At the adaptation stage, 25th February to 20th April, an optimum aeration level was tried to be found. In this period several times air supply decreased or increased in order to modify the results. After the adaptation stage, the condition was constant where nitrifiers and denitrifiers could grow in the same reactor. Consequently, the ammonium and nitrate concentrations were reduced simultaneously. The trend of concentration removal of ammonia is shown in figure 4-22.

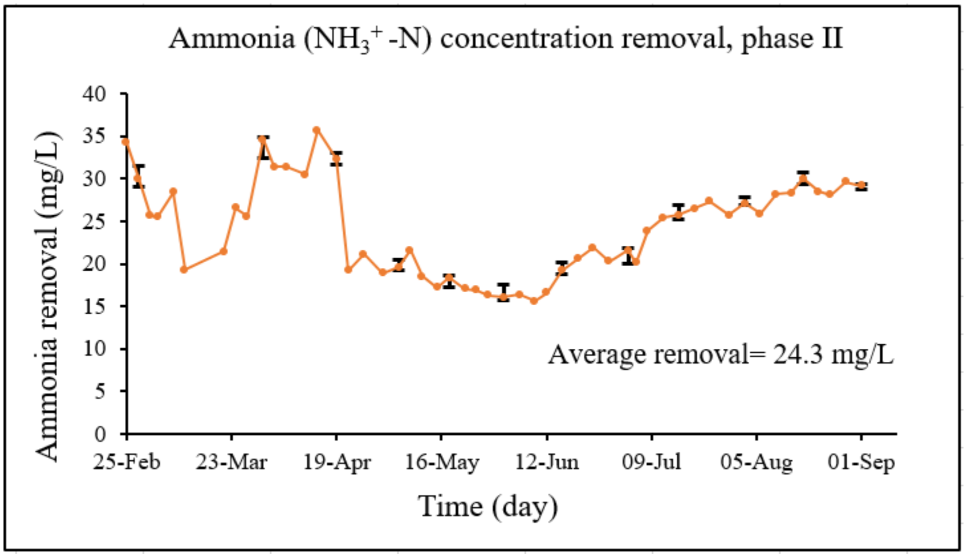


Figure 4-17 Ammonia-nitrogen concentration removal (mg/L) in phase II

The average ammonia-nitrogen concentration removal for the second phase was 24.3 mg/L and the removal efficiency was 61%. At the beginning of the second phase, the concentration removal was around 30 mg/L, which is similar to the last days of the first phase. The biomass and media were transferred from the IFAS reactor to the new EIFAS reactor, while the same DO was supplied. The ammonia-nitrogen removal varied between 15.6 mg/L and 35.7 mg/L and the removal efficiency changed from 39% to 96%.

In the second phase, the amount of air supplied to the reactor was lower than the first phase. Moreover, oxygen was consumed in close proximity to the cathode, while the electrical system was turned on (ON mode). In this phase, the aim was to remove ammonia and nitrate at the same time. For this reason, anoxic conditions were applied for this phase. Appendix A7 shows the detailed ammonia-nitrogen percentage removal in the second phase.

4.7.2 Nitrate-nitrogen, phase II

In order to remove nitrates as the product of ammonia oxidation in conventional nitrogen treatment process, an anoxic tank is added for denitrification after the aerobic digestion process. However, the novel idea of applying intermittent current ensures that both nitrification and denitrification are conducted in the same reactor. Thus, successful treatment depends on the balance between nitrification and denitrification.

In phase II of this study, the electrical field created a condition where it was expected to have a successful removal of all nitrogen compounds in a single tank. The nitrate-nitrogen concentration in the effluent is shown in figure 4-23. The average concentration of nitrate-nitrogen in the effluent was 9.8 mg/L and the concentration varied between 1.04 mg/L and 27.1 mg/L.

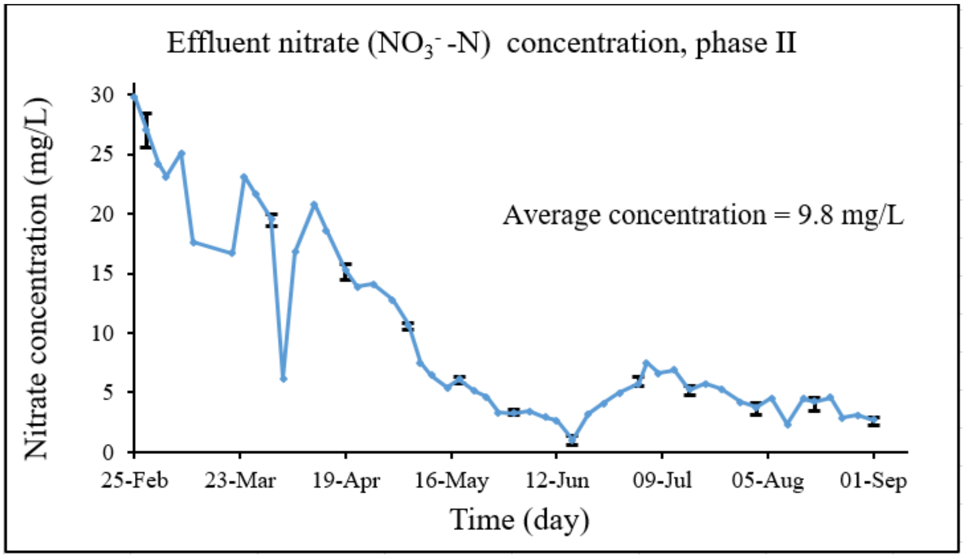


Figure 4-18 Nitrate-nitrogen concentration (mg/L) in the effluent in phase II

Less nitrate-nitrogen generation in the nitrification process and more nitrate-nitrogen consumption due to the stronger denitrification process are reasons that nitrate-nitrogen concentration decreased in the reactor. By contrast, in the first phase, under the higher DO level, a stronger nitrification process took place and nitrate concentration increased in the reactor over time.

4.7.3 Ammonia-nitrogen versus nitrate-nitrogen in enhanced EIFAS (phase II)

The changes in concentration of ammonia and nitrate indicate whether the sludge is under nitrification or denitrification process. A combination of the electrical system and Integrated Fixed-film Activated Sludge reactor would create a condition where both processes take place in the same reactor. A decrease of ammonia concentration and increase of nitrate concentration in the effluent can directly show the level of nitrification under oxidation conditions; similar to conventional bioreactors. Moreover, the simultaneous reduction of nitrate concentration and ammonia shows the level of denitrification in anoxic conditions.

When DO was not sufficient for the nitrification process, the ammonia removal and nitrate concentration were low (12th June). Ammonia-nitrogen concentration removal versus nitrate-nitrogen concentration in the effluent is shown in figure 4-24.

Results showed that when removal conditions for one form of inorganic nitrogen (ammonium, nitrate, nitrite) were optimal, TN removal was not optimum. This means that extreme nitrification or denitrification would create undesirable results. A comparison between the concentration of ammonia-nitrogen and nitrate-nitrogen could help to determine optimal treatment results. In order to remove TN, maintaining a balance between removal of ammonia and nitrate was required.

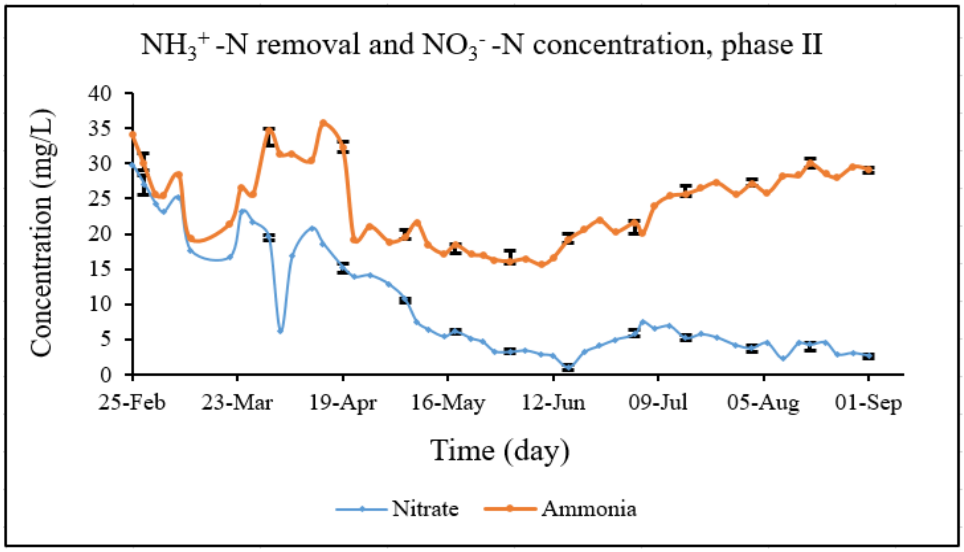


Figure 4-19 Ammonia-nitrogen concentration removal and nitrate-nitrogen concentration (mg/L) in phase II

Overall, most of the nitrate generated in phase I was discharged with the effluent without undergoing the denitrification. On the other hand, by applying the electrical field on the IFAS reactor, efficient denitrification took place. After July 1st, results became predictable; ORP was mostly negative at this stage.

4.7.4 Nitrate versus nitrite concentration (phase II)

Nitrite is produced in the ammonia oxidation process; then it is oxidized to nitrate to reach better stability. If sufficient oxygen is not available, nitrite accumulates in the reactor. Moreover, increase in nitrite concentration shows an anoxic condition in which nitrification takes place. However, the oxygen concentration is not high enough to prevent the denitrification process from taking place. Nitrite is essential to ignite the growth of other types of bacteria; for example Anammox bacteria. Therefore, measuring nitrite concentrations can help understand the state of the reactor. Figure 4-25 shows the nitrite concentration versus nitrate concentration in the second phase of the study.

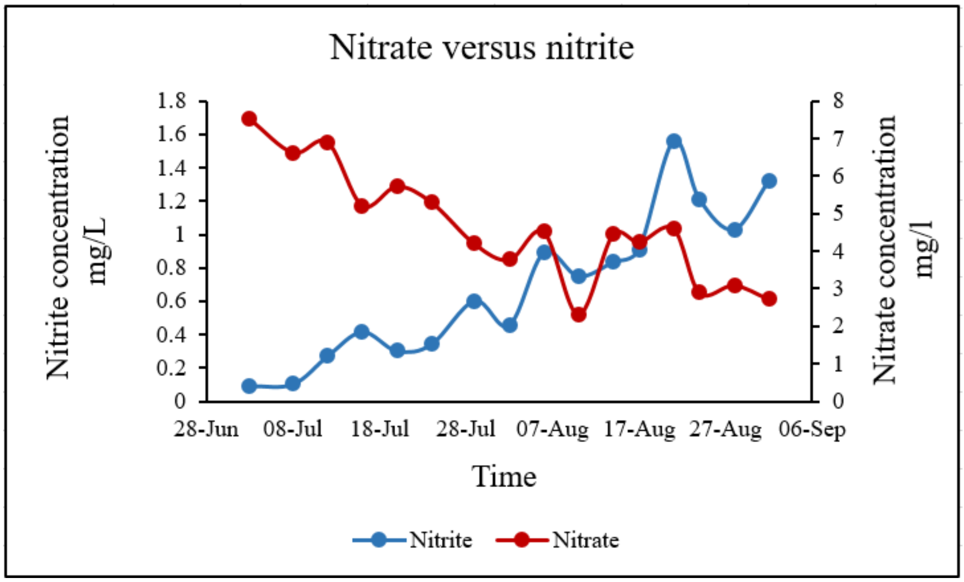


Figure 4-20 Nitrite concentration versus nitrate concentration (mg/L) for phase II

Nitrite concentrations increased in the second phase of the study, while nitrate concentrations declined. This was because of a drop in dissolved oxygen concentrations in anoxic conditions. The average nitrite concentration was 0.7 mg/L and the maximum concentration was 1.56 mg/L.

4.8 Phosphorus removal

Total phosphorus (TP) in wastewater includes inorganic soluble orthophosphates PO_4^{-3}-P, organic particulates and metal complexes (Park et al., 2011). In synthetic wastewater, only the soluble form of phosphorus was present since inorganic potassium phosphate was added to the mixture and the other two other forms of phosphor were not added to the synthetic wastewater. Therefore, analysis of phosphorus removal efficiency was based on that of orthophosphate results.

Most wastewater treatment plants use the coagulation process for removal of phosphorus. Aluminum or iron trivalent cations are added to the settling tank, where physicochemical reactions take place, and the phosphorus concentration decreases in the effluent. Reaction formulas are presented in equations (4-1) and (4-2). Conventional treatment methods cannot adequately remove phosphorus; only 2-5% of TP are absorbed by the biomass. Thus, extra treatment units or additional processes are required to remove remaining phosphorus.

$$Al^{3+} + (H_nPO_4)^{(3-n)-} \rightarrow AlPO_{4\,(p)} + nH^+ \qquad \text{Equation (4-1)}$$

$$Fe^{3+} + (H_nPO_4)^{(3-n)-} \rightarrow FePO_{4\,(p)} + nH^+ \qquad \text{Equation (4-2)}$$

4.8.1 Phase I

In the first phase of this study, orthophosphates were not removed since the conditions for the growth of phosphorus removal microorganisms were not provided. Moreover, chemical reactions like coagulation did not take place, since no additional coagulant was added to the reactor. For this reason, there was no expectation to remove phosphorus in this phase. Figure 4-26 shows the orthophosphate removal efficiency (in percentage) for the first phase.

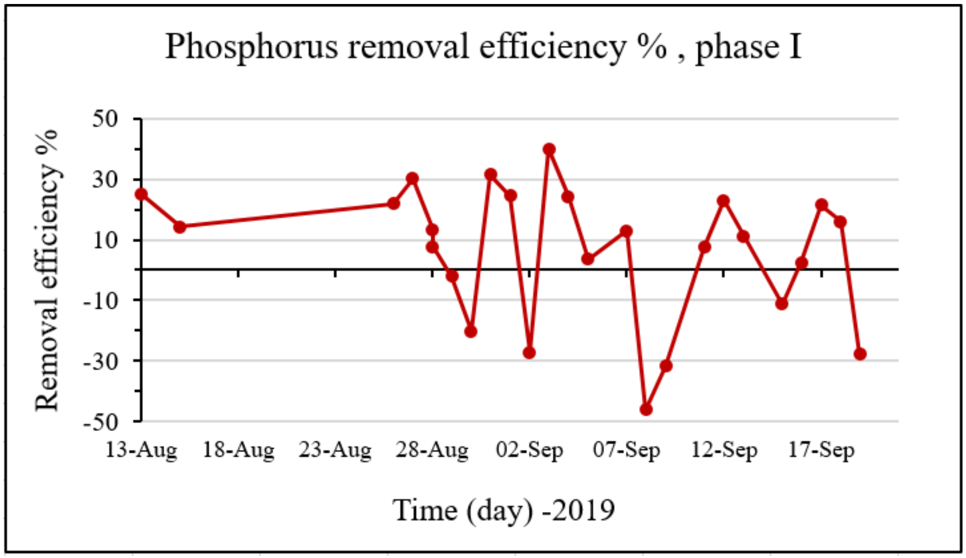


Figure 4-21 Orthophosphate removal efficiency in phase I

There were several points during this study, that orthophosphate removal efficiency was negative. This means that the effluent concentration was higher than the input concentration. Some days phosphorus concentrations were not reduced and all would accumulate in the reactor. The reason for this was that phosphorus would regenerate from dead cells as well as experiment errors.

The main phosphorus demand in this phase was because of microorganism cell generation. Normally, 1-5 mg/L of phosphorus was expected to be consumed by bacteria; this was seen in phase I.

4.8.2 Phase II

In the second phase of this study, high phosphorus removal efficiencies were recorded in the EIFAS reactor. Figure 4-27 shows orthophosphate removal efficiencies (in percent) in the second phase of the study.

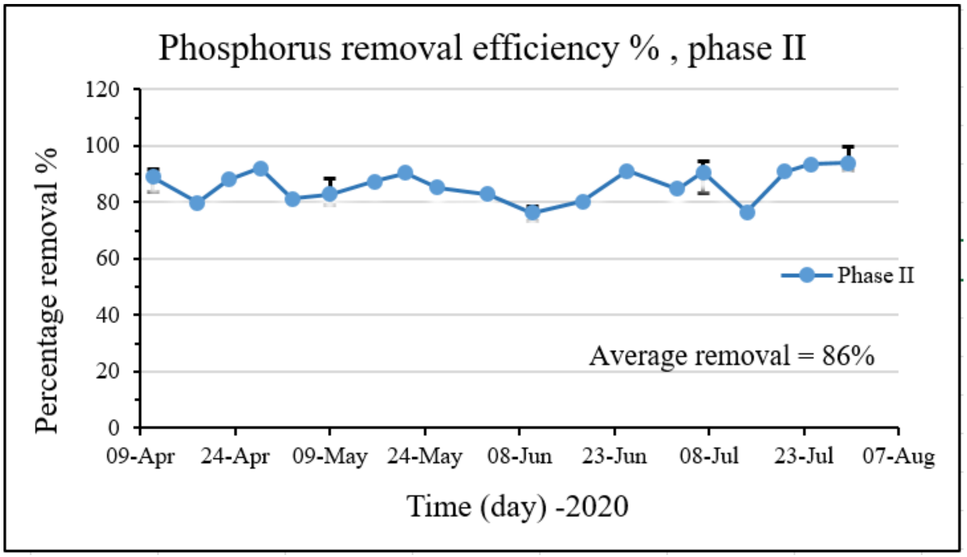


Figure 4-22 Orthophosphate removal efficiency in phase II

In the second phase, an average of 86% of orthophosphates were removed, and it increased to around 93% around the end of the study. These results show stable phosphorus removal, which indicate that electrocoagulation is a reliable approach used to remove the phosphorus in wastewater. Figure 4-28 shows influent and effluent concentrations of orthophosphates in the second phase of the study.

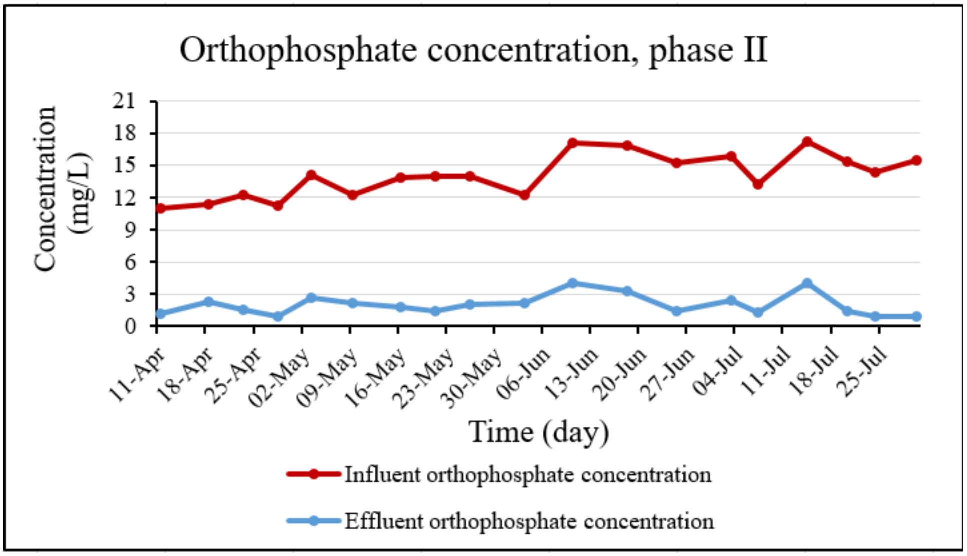


Figure 4-23 Influent and effluent orthophosphate concentration (mg/L) in phase II

Results show an average concentration removal of 12 mg/L, which reached 14.5 mg/L. Phosphorus compound can be removed using the electrocoagulation process. The aluminum released from the anode reacts to phosphates in the system, and aluminum phosphate precipitates as a result. Orthophosphate concentrations removed in phase II are shown in Appendix A6.

Assuming that the reactor discharge would be dumped in surface waters (rivers or lakes), TP concentrations should be regulated. The effluent had a flow rate of 25 L/day with an average concentration of 1.9 mg/L. Eutrophication caused by phosphorus discharge is a crucial problem for water bodies worldwide (Environment Canada, 2004).

Phosphorus concentration should not exceed 1 mg/L of PO_4-P, which is equal to 3 mg/L as phosphate (PO_4), and this limit was met in this phase. Tests were conducted adequate number times in order to ensure the validity of the results in the second phase.

4.9 COD removal

COD measurements in this study indicated the amount of carbon input as well as carbon released in the effluent. Carbon dissolved in influent and effluent were recorded and presented for each phase.

The desired influent COD was 350 mg/L. Variations of minimum and maximum COD inputs were caused by incomplete dissolution and chemical losses of carbon during the preparation of synthetic wastewater. Wastewater was sampled for further measurements in order to ensure the accuracy of the feedings.

Samples were taken from a depth of 10 cm from the feed tank containing synthetic wastewater. Measured COD concentrations changed after the start cycle of fresh synthetic wastewater. The maximum influent COD concentration fluctuation was 127 mg/L. Table 4-1 is the summary of COD treatment results in both phases.

Table 4-1 Influent and effluents COD concentration in phase I and phase II

Value	**COD influent (mg/L)**	**COD effluent (mg/L)**	**Removal (mg/L)**
Phase I			
Maximum	416	124	346.4
Minimum	289	7.02	225
Average	353	63	290
Phase II			
Maximum	419	109	385
Minimum	285	6.24	250.8
Average	345	35	310

The difference in wastewater COD concentrations may be a result of biological reactions in the system, water evaporation, or incomplete dissolution of glucose during the preparation of synthetic wastewater.

The EIFAS reactor was expected to remove nutrients in spite of COD fluctuations similar to the SMEBR response (Gao, 2014). The high organic carbon source in the influent is usually the reason for greater growth of microorganisms; organic carbon is used in the metabolism process. When carbon concentration is inadequate, slow reproduction and faster death rate reduce the number of microorganisms in sludge (Gao, 2014). In this study however, this scenario did not take place and an adequate amount of carbon was always provided to the system.

4.9.1 Phase I

Figure 4-29 shows the percentage of COD removal for the first phase of the study when Fixed-film was integrated with an Activated Sludge reactor (IFAS).

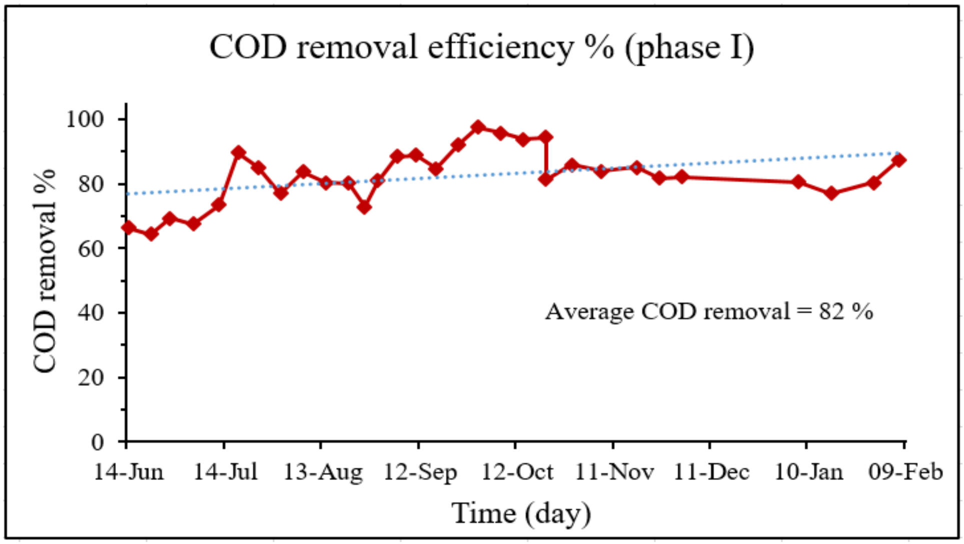


Figure 4-24 Chemical Oxygen Demand (COD) removal efficiency in phase I

In the first phase, average COD removal was 82%. COD removal fluctuated between 65% and 97%. The detail of the equation used to calculate the COD percentage removal is presented in the section A3 of appendix. Carbon oxidizing bacteria were the main consumer of carbon in phase I.

Moreover, concentration removal was significant because influent COD concentrations changed. Microorganisms' carbon intake was indicated rather by concentration removal and influent COD concentrations directly would not affect it. In the first phase of the study, average COD removal increased to 289 mg/L. The detail of all concentration measurements for the COD removal is presented in section A4 of the appendix.

4.9.2 Phase II

The second phase of the study evaluated the efficiency of an Electro Integrated Fixed-film Activated Sludge reactor. The media used in the IFAS reactor was transferred to the EIFAS reactor which biofilm growth was initiated on it. The COD removal efficiency increased to 99% in phase II and average removal was 90%. Figure 4-30 shows the COD removal in percentage in phase II of this investigation. The COD removal in mg/L in phase II is shown in appendix A5.

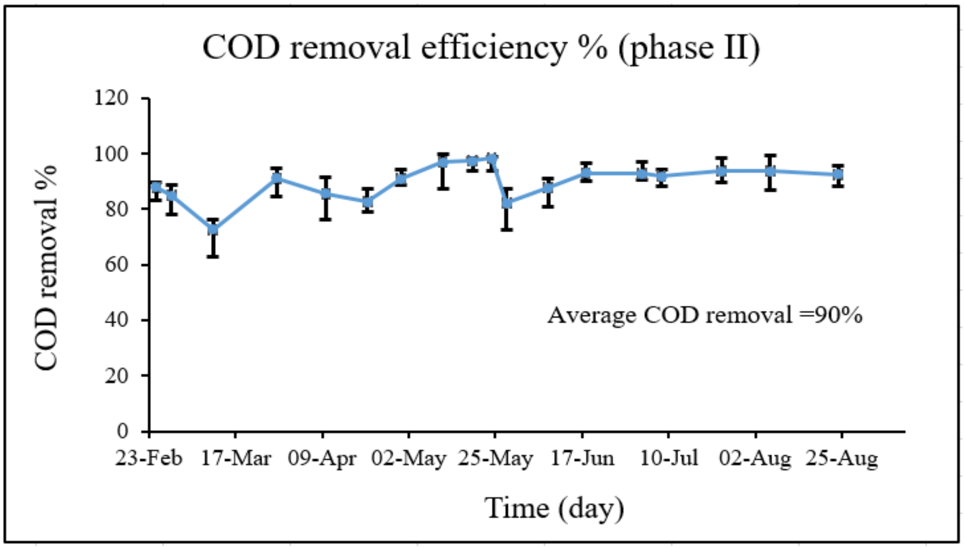


Figure 4-25 Chemical Oxygen Demand (COD) removal efficiency in phase II

Electrical current increases COD removal in the bioreactor (Hasan et al., 2014). The COD removal in SMEBR could reach 90% to almost 100% when sufficient C/N ratio was provided to microorganisms (Gao, 2014). In the same study, the result showed that insufficient carbon input affected the COD removal (Gao, 2014).

Average COD concentration removal was 310 mg/L; it was 20 mg/L more than the COD removal in phase I. The maximum removal was 390 mg/L. Due to the change in air supply, on the 25th of May, there was a decline in the COD removal, which was recovered overtime.

COD removal in the EIFAS reactor was better than IFAS reactor. The increase in carbon consumption was because of the electrocoagulation of colloidal carbon. Moreover, carbon oxidizers, heterotrophic denitrifiers and PAO were more active; all consumed carbon to survive and breed.

4.10 Discussion summary

Promising nitrification process occurred in the Integrated Fixed-film Activated Sludge reactor when an adequate oxygen was provided to the reactor. The biofilm growing on the plastic media contained several types of bacteria especially nitrifying bacteria. The first phase of the study was conducted under extremely aerobic conditions with average dissolved oxygen of 7 mg/L.

Therefore, nitrifying bacteria were dominant because of the high concentration of ammonia in the influent and air injection to the reactor. It was expected that nitrifiers grow on the outer layer of plastic media where microorganisms directly contact to oxygen. Besides, denitrifying bacteria were active at the bottom of the reactor and inner layers of media where anoxic and anaerobic conditions took place.

Electro Integrated Fixed-film Activated Sludge reactor was operated in a lower dissolved oxygen level in the second phase of the study for six months. The electrical field, by providing intermittent oxidation and reduction states in the reactor allows both nitrifying and denitrifying bacteria to grow and breed in the same reactor. Figure 4-31 shows the development of biofilm on the media surface over time. More biofilm was generated on media when it stayed for longer time in the reactor; the growth of biofilm changed the colour of media over time.

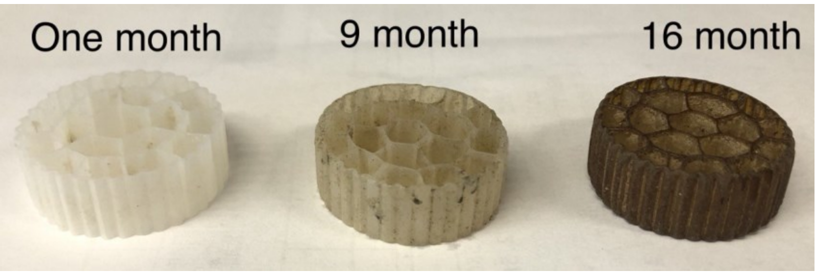


Figure 4-26 Media biofilm development throughout the study

Table 4-2 shows a summary of overall results in two phases of the study. Phase I lasted for ten months, whereas phase II lasted for six months.

Table 4-2 Summary of overall results (phase I and phase II)

	Phase I	**Phase II**
Ammonia-nitrogen removal (mg/L)	39	30
Nitrate-nitrogen concentration (mg/L)	30	2
Orthophosphate removal %	-	93
COD removal %	90	98

Chapter V

Conclusions, Contribution and Future work

5.1 Conclusion

This study was conducted in two phases in order to prove that the novel Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS) enhances nitrogen removal through the use of electrokinetic control of redox conditions in a single vessel.

Phase I of the study focused on ammonia removal in an Integrated Fixed-film Activated Sludge reactor (IFAS) under aerobic condition with an average dissolved oxygen (DO) of 7 mg/L. Despite conventional methods, the IFAS reactor worked under a low concentration of biomass. The results from this phase of the study showed a promising nitrification process in IFAS in aerobic condition after ten months.

A high-efficiency ammonia-nitrogen removal of about 96% was attained from the influent with high ammonia concentration; even though an unwanted increase in nitrate concentration was recorded. The nitrification capacity of the reactor, which depended on adequate DO levels and ammonia input, had increased over the course of this experimental study. Two comparative reactors, consisting of a fixed-biofilm reactor (media reactor) and an activated sludge reactor (AS) assessed the effect of the attached culture on ammonia removal.

Six weeks after starting the phase I, the media reactor system was able to remove about 10.3 mg/L of ammonia-nitrogen. The amount of nitrate-nitrogen increased to 6.55 mg/L in the same time frame. After twenty-nine weeks, the ammonia-nitrogen removal improved to 23.5 mg/L, while nitrate-nitrogen concentration reached 13.6 mg/L. Simultaneously, the ammonia removal in the AS reactor elevated from 6.55 mg/L after six weeks to 13.8 mg/L after twenty-nine weeks. Stated results show that the attached culture system was effective in the ammonia removal and higher than the suspended culture reactor. Because the SRT for biofilm growing on media was longer than the activated sludge, biofilm actively participates in the nitrification process. Therefore, nutrient removal was maintained, even without suspended biomass in the reactor.

In the second phase, a variation of aerobic and anoxic conditions due to controlled ORP values, which these values fluctuated between 92 mV and -189 mV, showed the significant ammonia-nitrogen removal. Moreover, low nitrate-nitrogen concentrations were recorded in the Electro-Integrated Fixed-film Activated Sludge reactor (EIFAS). Six months after the second phase of the study began, 29.2 mg/L of ammonia-nitrogen was removed, and 2.7 mg/L of nitrate-nitrogen concentration were logged. Furthermore, the EIFAS reactor showed successful removal of orthophosphate and chemical oxygen demand (COD), which reached 93% and 98% respectively.

The novel EIFAS system removed nutrient (ammonia, nitrates and phosphorous) contents as well as COD from high concentrated influent in a single vessel. EIFAS system is particularly efficient for testing wastewater containing a high amount of ammonia. The technology follows sustainable development principles by decreasing the footprint and energy consumption. It can be used in small and medium-sized wastewater treatment plants, upgrading existing facilities in order to prevent eutrophication of water receptors.

5.2 Contribution

This study developed and tested an Electro-Integrated Fixed-film Activated Sludge, which enhanced wastewater treatment due to combination of activated sludge, biofilm, and electrokinetic phenomena.

This work also defined the contribution of all the Integrated Fixed-film Activated Sludge components in nutrient removal.

Moreover, this study introduced a novel design for the EIFAS reactor on a lab-scale and verified the solidity and efficiency of such design based on the recorded results in a long-term.

5.3 Future work

Results of this study supposed to be applied to a pilot-scale wastewater treatment system with real wastewater inflow in order to further confirm these results. There are many differences between small and large-scale reactors, particularly with respect to of DO concentration, current value, and sludge quality control. This study showed that control of current density and DO concentration must be considered in the scale-up process.

In order to obtain better understanding of the species and types of microorganisms as well as other settled particles on media surfaces, micro-scale analyses are required.

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Appendix

A1 Calculation of percentage removal

To calculate the percentage of COD or ammonia-nitrogen removal, underneath formula was applied.

$$R\text{emoval percentage} = \frac{\text{Inflow concentration} - \text{Measured concentration}}{\text{Measured concentration}} * 100$$

Where inflow concentration is equal to the ammonia-nitrogen concentration in the synthetic wastewater, mg/L NH_3^--N; and measured concentration is the ammonia-nitrogen concentration in the effluent sample, mg/L NH_3^--N.

A2 Calculation of percentage of phosphorus removal

To calculate the percentage of phosphorus removal the following formula was conducted.

$$\text{Phosphorus removal percentage} = \frac{\text{Inflow concentration} - \text{Measured concentration}}{\text{Measured concentration}} * 100$$

Phosphorus inflow is the influent phosphorus concentration and measured concentration is the phosphorus concentration in the effluent sample, mg/L PO_4^--P.

A3 Calculation of percentage of COD removal

To calculate the percentage of COD removal the following formula was conducted.

$$\text{COD percentage removal} = \frac{\text{Inflow concentration} - \text{Measured concentration}}{\text{Measured concentration}} * 100$$

COD inflow is the influent COD concentration and measured concentration is the COD concentration in the effluent sample, mg/L of COD.

A4 COD concentration removal for the first phase of the study

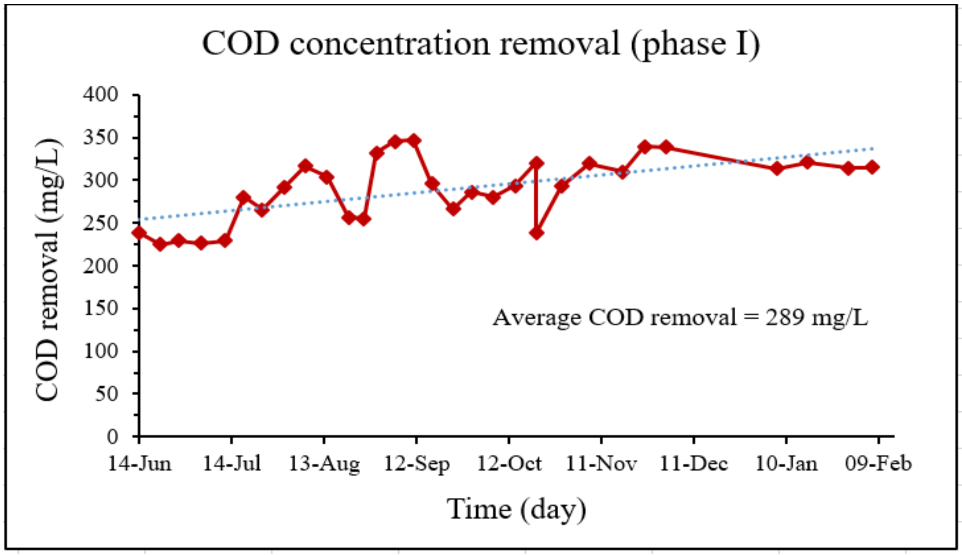

Figure A-1 Chemical Oxygen Demand (COD) concentration removal for phase I

A5COD concentration removal for the second phase of the study

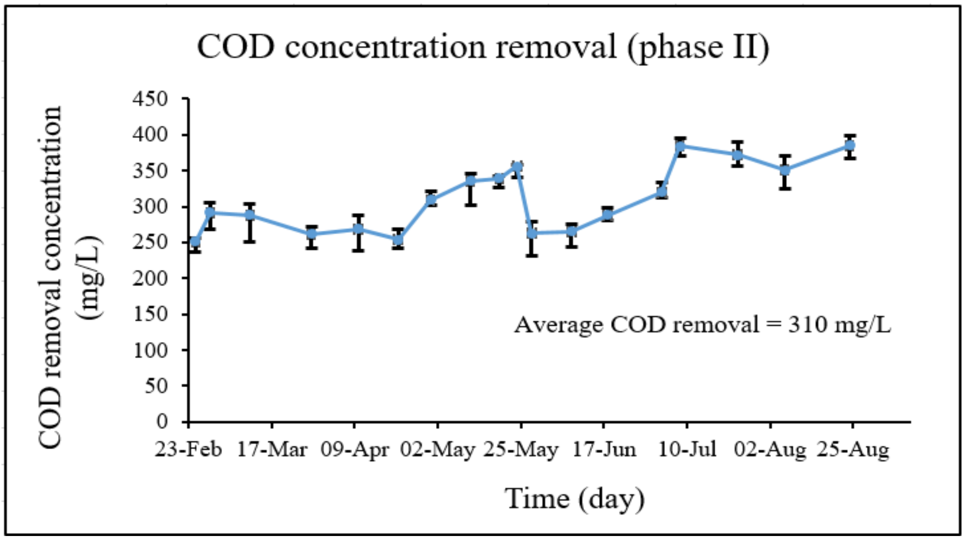

Figure A-2 Chemical Oxygen Demand (COD) concentration removal for phase II

A6 Phosphorus concentration removal for the second phase of the study

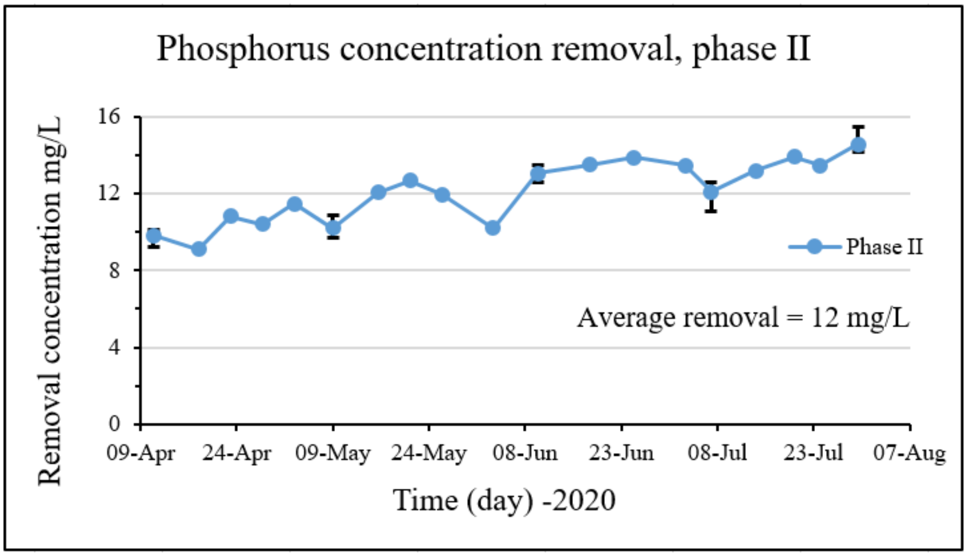

Figure A-3 Orthophosphate concentration removal (mg/L) for phase II

A7 Ammonia-nitrogen percentage removal for phase II

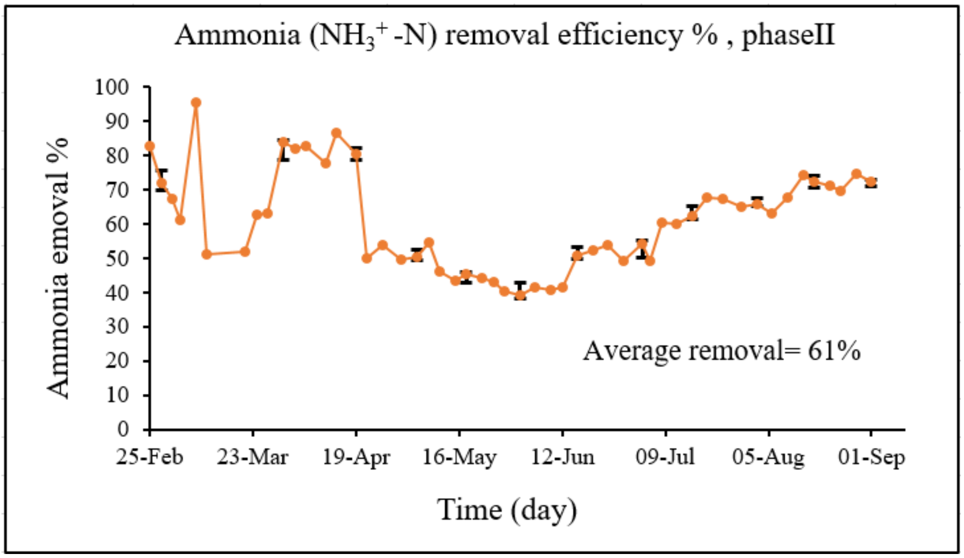

Figure A-4 Ammonia-nitrogen percentage removal for phase II