Upconcentration of Domestic Wastewater through Biosorption Sedimentation Process

Submitted by

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List of Abbreviations

ABF	Air Back Flushing
AD	Anaerobic Digestion
AS	Activated Sludge
ASM	Aerated Sewage Microfiltration
BSP	Biosorption-Sedimentation Process
C:N:P	Carbon To Nitrogen To Phosphorus Ratio
CCM	Combined Coagulation Microfiltration
CCM	Combined Coagulation Microfiltration
CEPT	Chemically Enhanced Primary Sedimentation
CF	Concentration Factor
COD	Chemical Oxygen Demand
DSM	Direct Sewage Microfiltration
DSM	Direct Sewage Microfiltration
E-MCR	Enhanced Membrane Coagulation Reactor
g bCOD g VSS $^{-1}$ d^{-1}	Grams Of Biodegradable Chemical Oxygen Demand Per Gram Of Volatile Suspended Solids Per Day
HCM	Hybrid Coagulation Microfiltration
HiCAS	High Rate Conventional Activated Sludge
HiCS	High Rate Contact Stabilization
HL-MBR	High Loaded Membrane Bioreactor
HL-MBR	High Loaded Mbrs
HRAS	High-Rate Activated Sludge
HRT	Hydraulic retention Time

IE	Ion Exchange
IESE	Institute Of Environmental Sciences & Engineering
MFCs	Microbial Fuel Cells
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
MPC	Membrane-Based Pre-Concentration
MPC	Membrane-Based Pre-Concentration
NH_4^+-N	Ammonium-Nitrogen
NO_3^{-1}–N	Nitrate - Nitrogen
OM	Organic Matter
PAOs	Phosphorous Accumulating Organisms
PCRWR	Pakistan Council Of Research In Water Resources
R^2	Regression Co-Efficient
RPM	Revolutions Per Minute
RSM	Response Surface Methodology
SRT	Solids Retention Time
T_{ano}/T_{an}	Anoxic To Anaerobic HRTs
TCOD	Total Chemical Oxygen Demand
TKN	Total Kjeldahl Nitrogen
TP	Total Phosphorus
UNEP	United Nations Environment Programme
UN-HABITAT	United Nations Human Settlements Programme
VFAs	Volatile Fatty Acids

Abstract

This study demonstrates a feasible and novel design of up-concentration of domestic sewage through Biosorption-Sedimentation process (BSP) for maximum energy production via anaerobic digestion in later stage. System was operated at various anoxic to anaerobic HRTs (T_{ano}/T_{an}) with an infinite SRT to obtain up-concentrated stream (in terms of COD and Total Phosphorous) and minimum ammonium-nitrogen concentrations in effluent. Results indicate that the potential of net energy yield up to 15.5 kWh/m^3 at could be achieved an OLR of 12 kg of COD/m^3/d. T_{ano}/T_{an} of 60 minutes/60 minutes was selected as optimum ratio, keeping in view the maximum potential energy in terms of COD, Carbon: Nitrogen: Phosphorous (C:N:P) ratio and ammonium-nitrogen concentration of effluent. This study concludes that BSP may efficiently overcome the issue pertaining to sludge treatment and disposal associated with functional aerobic wastewater treatment plants. Also maximum energy can be extracted via anaerobic digestion of concentrated stream due to an addition of up concentration step in future treatment plants in order to make net energy positive wastewater treatment plants.

Chapter 1

Introduction

1.1 Background

The volume of wastewater generation increases directly with an exponential increase in world’s population, improved living standards of people, urbanization and also due to economic development (Benetti, 2008). In order to address this global issue, wastewater treatment plants have been developed all over the world, not merely to treat wastewater but also to reuse the treated wastewater/effluent for non-portable purposes.

By far the most unpredictable factor at present is the effect of future climate change. Although this might require major infrastructural works and management schemes, technological positivism should allow us to provide better sanitation to more people with a focus on neutral energy or net positive wastewater treatment technologies (Hillman et al., 2008)

It has been reported that almost 330 km^3/year of municipal wastewater generated worldwide, that is theoretically supposed to be enough for the production of biogas for millions of households and to irrigate millions of hectares of agriculture land, in particular of crop yield (Mateo-Sagasta et al., 2015).

Out of 6.849 km^3/year of municipal wastewater generated in Pakistan, only 0.548 km^3/year has been treated making it less than 8 % of total wastewater generated (Sato et al., 2013). Recently, Pakistan Council of Research in Water Resources (PCRWR) reported that less than 10% of urban wastewater is treated in Pakistan (Raza et al., 2017).

1.2 Problem Statement

Municipal wastewater has been characterized by low organic strength and a high fraction of suspended and colloidal organic matter, making the direct anaerobic sewage treatment process as uneconomical because of the low organic loading in untreated wastewater. An appropriate pre-concentration step should be implemented for maximum energy recovery/production directly from the sewage organics.

1.3 Objectives of the study

- Maximize COD and Total Phosphorous (TP) concentration of medium-strength wastewater.
- Minimize Ammonium-Nitrogen (NH_4^+-N) concentration of the wastewater.
- Optimization of Anoxic to Anaerobic HRT ratio (T_{ano}/T_{an}) with respect to C:N:P ratio for maximum biogas potential.
- Optimization of Anoxic to Anaerobic HRT ratio (T_{ano}/T_{an}) of the system by using Response Surface Methodology (RSM) software.

➢ Chapter 2

Literature Review

Municipal wastewater is now considered as a resource to make treatment plants as energy self-sufficient. Organics within the wastewater have a potential to produce bio-gas, if digested anaerobically (Leitao et al., 2006). Wastewater production increased with an increase in population, putting stress on existing water sources. Also there exist a huge potential to recover energy from domestic organics that could ends up with energy self-sufficient treatment plant.

Several candidate up-concentration technologies have been proposed like conventional activated sludge (CAS), high rate contact stabilization (HiCS), different membrane based technologies like enhanced membrane coagulation reactor (E-MCR), membrane-based pre-concentration (MPC), combined coagulation microfiltration (CCM), direct sewage microfiltration (DSM) and continuous aerated sewage microfiltration (ASM), High Loaded Membrane Bioreactor (HL-MBR), centrifugation, filtration and especially the chemically enhanced primary sedimentation (CEPT) system (Khararjian and Smith, 2008) for the production of biogas via anaerobic digestion.

2.1 Conventional Activated Sludge (CAS) process

Activated sludge (AS) systems are commonly used for robust and efficient treatment of municipal wastewater. However, these systems cannot achieve their maximum potential to recover valuable resources from wastewater (Khiewwijit et al., 2015). Besides purification, resource recovery is gaining importance as a main purpose of CAS process. This inspires a search for technologies that not only remove resources like organic matter, nitrogen and phosphorous from the municipal wastewater, but make them available for reuse (Meerburg et al., 2015). A typical CAS process requires a net annual energy input in the order of 40 kWh per population equivalent (PE) (Zessner et al., 2010).The caloric energy content of raw domestic sewage exceeds the electricity demand to operate a CAS plant by at least a factor of nine (Meerburg et al., 2015). Conversion of caloric energy from wastewater organics to useful forms of energy is most often achieved via methane production during anaerobic digestion. Therefore, pre-concentration of domestic wastewater allows to produce a more concentrated organic stream as sludge, and allows efficient side stream anaerobic digestion (Verstraete and Vlaeminck, 2011). More concentrated stream results in more energy recovery and ultimately make possible to achieve net energy-neutral or even energy-positive wastewater treatment (Jenicek et al., 2013). A typical CAS with excess sludge treatment is shown in **Figure** 1.

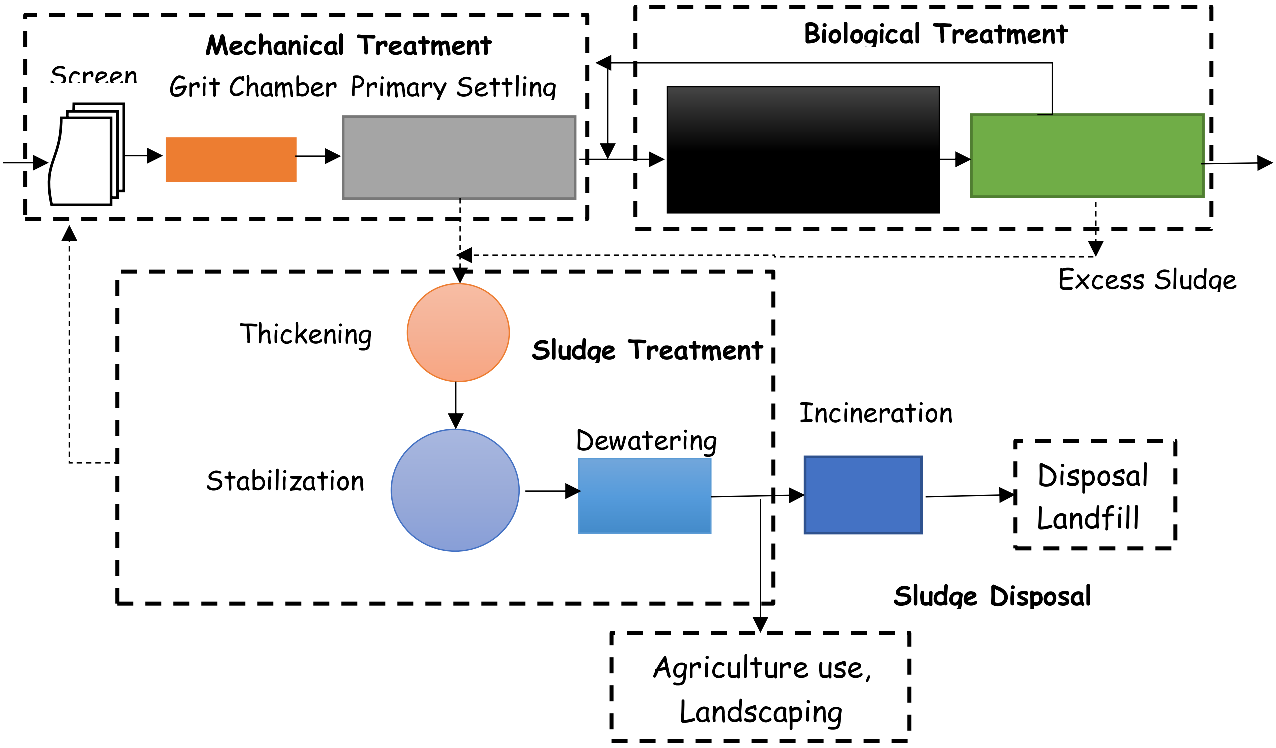


Figure 1: A Typical Conventional Activated Sludge Process **(Jenicek et al., 2013)**

However, high coagulant and flocculants costs, and their suspected inhibition of anaerobic conversion processes are serious disadvantages. In addition, side products from synthetic polymers can be toxic to humans (Salehizadeh and Shojaosadati, 2001). Also, direct micro or ultrafiltration of municipal wastewater is possible. However, these membrane processes suffer from severe membrane fouling with associated high energy consumption (Al-Malack and Anderson, 1997). A better option would be bio flocculation of the sewage organic suspended and colloidal matter, followed by settling or membrane filtration (Faust et al., 2014; Leal et al., 2010). In particular the combination of bio flocculation and membrane filtration could be attractive because this not only concentrates the organic matter, but also produces a nutrient containing and particle free effluent, which can be used for irrigation (Muston et al., 2005). **Figure**. 2 depict how the bio flocculation of sewage organics can be done along with membrane treatment of the sewage.

The LIFE NECOVERY project aims to demonstrate, by means of a prototype, the feasibility of an innovative WWTP flow sheet based on a Pre-concentration step at the inlet of the WWTP and focused on the recovery of nutrients and energy (You Chen, 2018). The innovative and crucial step is the pre-concentration (biosorption) which produced an upper effluent with very low solids and a bottom effluent with high quantity of solids leading to a higher biogas production. The upper diluted stream was treated in a zeolite adsorption unit in order to recover the nitrogen; the lower stream (enriched sludge) was treated in a conventional Anaerobic Digestion unit in order to obtain energy from the biogas formed. 70% of nutrients (Nitrogen and Phosphorous) was recovered, 80% by-products return to land by reusing the sludge produced in Anaerobic Digestion and 30% reduction in carbon footprints (Liu et al., 2009).

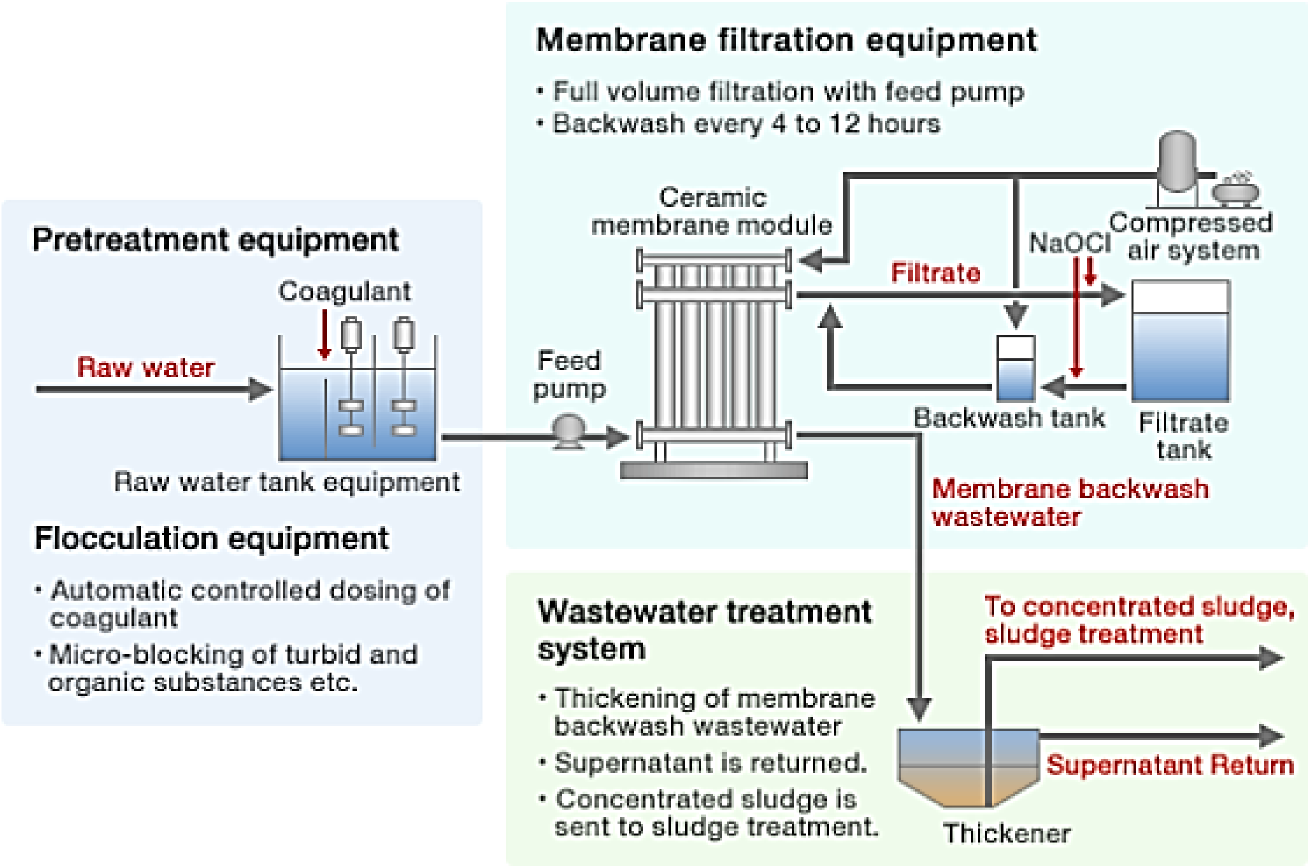


Figure 2: Bio flocculation and Membrane Treatment of Sewage (Muston et al., 2005)

2.2 High rate Conventional Activated Sludge process

In recent years, high rate conventional activated sludge process has been introduced in order to remove maximum organics from wastewater stream and to produce more sludge. Sludge produced in sedimentation tank was recirculated to contact tank. Removal efficiency of organics was reported in the range of 50-70% at shorter HRT of contact tank (less than 30 minutes). For achieving this purpose, short HRT of contact tank allowed rapid removal of organics from wastewater. Diamantis et al. (2013) reported the sludge specific loading rate of HiCAS process was in the range of 2-10 grams of biodegradable chemical oxygen demand per gram of volatile suspended solids per day (g bCOD g VSS $^{-1}$ d^{-1}) and a very short sludge retention time (SRT) ranging from several hours to two days. Meerburg et al. (2015) reported a high potential of energy recovery from anaerobic digestion of produced sludge at shorter SRT in HiCAS in order to approach net energy-neutral wastewater treatment process.

2.3 High-rate Activated Sludge System

For making wastewater treatment as an energetically self-sufficient, there is a need to concentrate wastewater organics (chemical energy) to achieve a high efficiency in methane production via direct anaerobic digestion. Therefore, a promising technology is the high-load contact stabilization (HiCS) system. This is a high-rate activated sludge (HRAS) system, an initial stage of two-stage activated sludge system. High loading rates i.e. greater than 2 g bCOD g^{-1} VSS d^{-1}, short sludge retention times (SRT) i.e. less than 3 days and feast-famine regime are considered as an important characteristics of high rate activated sludge (HRAS). Moreover, feast-famine aids in selection of microorganisms with fast biosorption and bio-accumulation abilities. Experiments revealed that sludge was produced in the range of yield 0.737 - 0.438 kg VSS kg^{-1} COD $_{removed}$ against SRT of 0.46-2.82 days, respectively at constant contact to stabilization time of 15:40 min , and the highest net recovery of organics.

2.4 High-rate Contact Stabilization process

Another study on high-rate contact stabilization (HiCS) reactor was performed, with high sludge-specific loading rates (>2 kg bCOD kg^{-1} TSS d^{-1}) and low sludge retention times (<1.2 d) (Meerburg et al., 2015). Study revealed that 36% of influent chemical energy was recovered in high-rate contact stabilization (HiCS) system as methane through anaerobic digestion with less amount of CO_2 production and more sludge yield as compared to high-rate conventional activated sludge (HiCAS) process. COD removal rates were always significantly lower in the low-rate reactors than in their respective high-rate counterparts (Meerburg et al., 2015). **Figure** 3 shows a comparison between CAS, HiCAS, CS and HiCS processes.

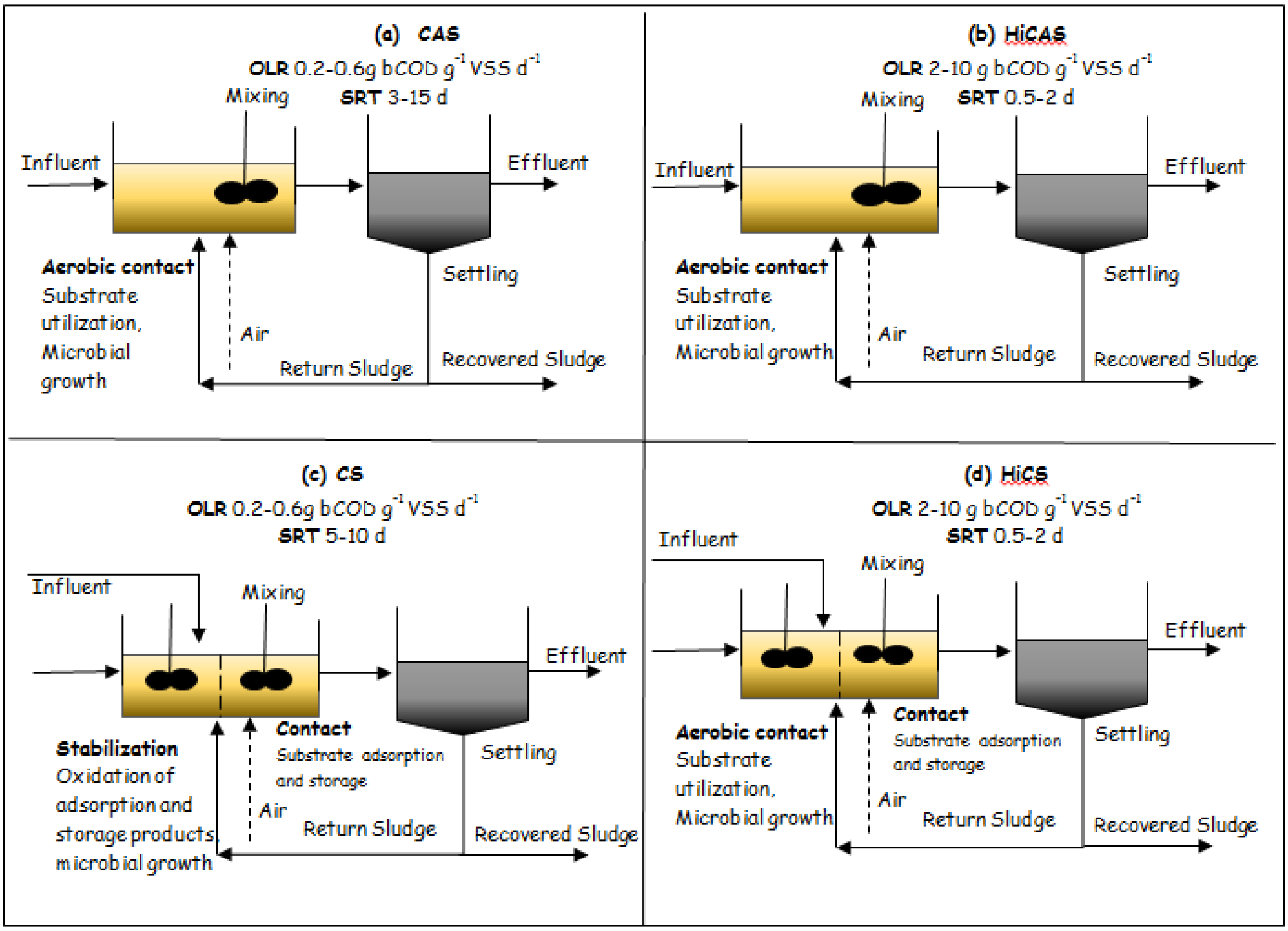


Figure 3: A Comparison among CAS, HiCAS, CS and HiCS Processes (Meerburg et al., 2015)

2.5 Chemically Enhanced Primary Treatment (CEPT)

However, neither the physical methods nor Chemically Enhanced Primary Treatment (CEPT) are optimized for removal of dissolved organic matter. On the contrary, using an advanced concentrator at a high sludge loading rate, where biosorption and bio-accumulation become important processes, can concentrate the particulate and colloidal fraction as well as the soluble fraction of wastewater. Biosorption is defined as the physiochemical process that passively concentrates and binds organic matter onto the biomass. Bio-accumulation on the other hand is the active metabolic process to absorb organic matter onto and within the biomass driven by the respiration energy of the microorganisms (Pauwels, 2015). A typical layout of CEPT process is shown in **Figure** 4.

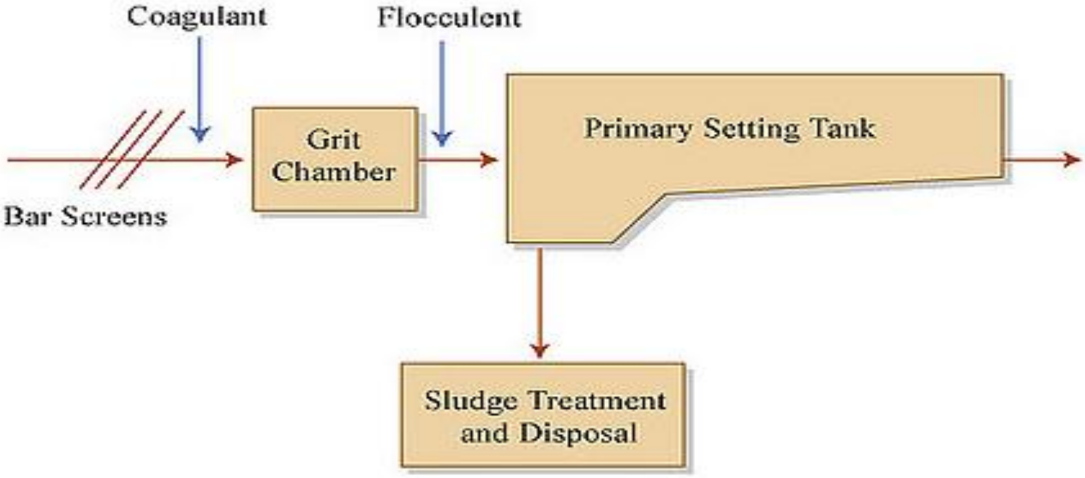


Figure 4: Process Flow Diagram of CEPT process

Another possibility that has been proposed as a candidate technology to achieve energy-neutral wastewater treatment is CEPT, followed by anaerobic digestion of the produced sludge (Diamantis et al., 2013). Typically CEPT with an optimal concentration of Al^{3+} as a coagulant, achieves COD $_{part}$ removal efficiencies of 85 % (Tchobanoglous et al., 1991). Therefore, neither the physical separation methods nor CEPT are optimized for removal of dissolved organic matter, which limits the maximum amount of energy that can be recovered and leaves a considerable fraction of organics to be treated in subsequent stages (Meerburg et al., 2015). Concentration of organic matter may also be achieved by primary sedimentation or by chemically enhanced primary sedimentation (CEPT), which achieve a particulate matter removal of typically around 60% and 85%, respectively (Tchobanoglous et al., 1991).

2.6 Membrane-based Technologies

2.6.1. High-loaded Membrane Bioreactor

Leal et al. (2010) proved that it is feasible to concentrate grey water up to 10 times its original COD value with a high-loaded membrane bioreactor (HL-MBR). During bio-flocculation microorganisms partly consume soluble biodegradable pollutants and excrete polymers that induce flocculation of colloidal and suspended wastewater particles. Because in this manner smaller particles aggregate into bigger particles, membrane fouling is considerably reduced compared to direct membrane filtration (Ivanovic et al., 2008). When operated at very short sludge retention times (SRT, typically 0.1-0.5 d), in combination with very short hydraulic retention times (HRT, typically below 1h), high concentrations of organic matter can be produced while (aerobic) mineralization of organic matter can be minimized to less than 10% (Faust et al., 2014). High loaded MBRs (HL-MBR) studied the effect of SRT on the extent of bio-flocculation. It was reported that fraction of suspended COD in the concentrate increased from 59 to 98% as SRT increased from 0.125 d to 5 d. The loss of sewage organic matter as a result of biological oxidation was ranging from 1-32% at SRT of 0.125-5 d, respectively. At a longer SRT i.e. 5 d, maximum mineralization was reported (32%). Shorter SRT result in more membrane fouling, which can be due to poor bio-flocculation and high submicron particle concentrations (Faust et. al, 2014). **Figure** 5 depicts HL-MBR combined with anaerobic digester (AD) for the production of methane.

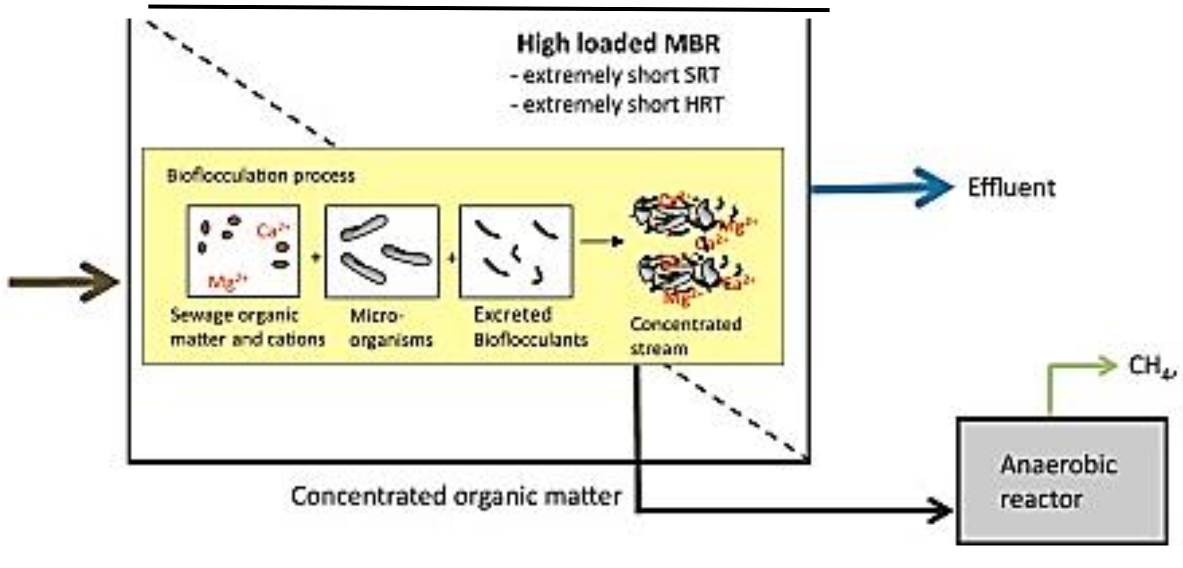


Figure 5: High Loaded MBR couple with AD (Faust et. al, 2014)

2.6.2. Enhanced Membrane Coagulation Reactor

Only few studies have examined the role of coagulation in the sewage up-concentration process. (Gong et al. (2014) reported that use of more coagulants resulting in solutes deposition on the surface of membrane, ultimately suppressed its resistance. Coagulant like polyaluminum chloride (PACl), enhanced flock formation at their optimum dosage in coagulation step; averting irreversible fouling by removing foulants and colloids within 6 days. Therefore, coagulation in the upconcentration process is considered as inefficient as organic matter retains, demanding the addition of other treatment materials (Odey et al., 2017). An appropriate and alternative approach, is the use of coagulants and adsorbents, like polyaluminum chloride (PACl) and powder-activated carbon (PAC) capable of achieving high concentration efficiency and minimizing membrane fouling. Addition of powder-activated carbon (PAC) and polyaluminum chloride (PACl) in enhanced membrane coagulation reactor (E-MCR), enhanced the concentration efficiency of sewage organics ranging between 6000 to 9800 mg/L of chemical oxygen demand (COD) and also minimizing membrane fouling, too. The reactor was operated for 100 days with 70 days without severe fouling. The concentrate was used as feed for AD using a continuous stirred tank reactor. The result shows that biogas yields (in mL/g COD) enhanced directly with HRT (in days) of the anaerobic digester at mesophilic temperature (35^{o}C). Therefore making E-MCR as a promising option for sewage concentration and energy recovery (Odey et al., 2017).

2.6.3. Membrane-based pre-concentration coupled with Ion Exchange process

This study proposes the recovery of organics and nitrogen from sewage through membrane-based pre-concentration (MPC) combined with ion exchange (IE) process. Despite the low rate of recovery, the process could achieve a total of 0.38 kilo-Watts hour per cubic meter (kWh/m^3) energy recovery by combining energy production with anaerobic digestion of pre-concentrated organics (0.26 kWh/m^3) and energy saving via nitrogen reuse (0.12 kWh/m^3). Unlike conventional activated sludge process, MPC–IE redesigned organic carbon flow to increase chemical oxygen demand (COD) conversion for energy recovery via anaerobic digestion (AD). This process also achieved nitrogen recovery instead of destruction.

Membrane-based pre-concentration of COD recovered up to 65% of COD and IE recovered 37.5% of ammonium-nitrogen (NH_4^+-N). This process enhanced energy recovery due to increased COD through anaerobic digestion (AD) and nitrogen recovery instead of destruction from sewage. The MPC–IE process redesigned carbon flow during sewage treatment. Organic COD in the form of concentrated state for AD energy production was essential for pursuing energy neutrality in WWTPs. High COD pre-concentration (higher than 65%) indicates the potential of COD pre-concentration as a self-sufficient energy process. The amount of energy saved could reach a remarkable level (0.50 kWh/m^3 for sewage in this study assuming 100% influent NH_4^+-N recovery). **Figure** 6 shows a typical configuration of membrane based pre-concentration coupled with Ion Exchange process. The MPC–IE process increased energy savings via N reuse, which is not considered in the typical CAS process. The amount of energy saved could reach a remarkable level (0.50 kWh/m^3 for sewage in this study assuming 100% influent NH_4^+-N recovery) (Gong et al., 2017).

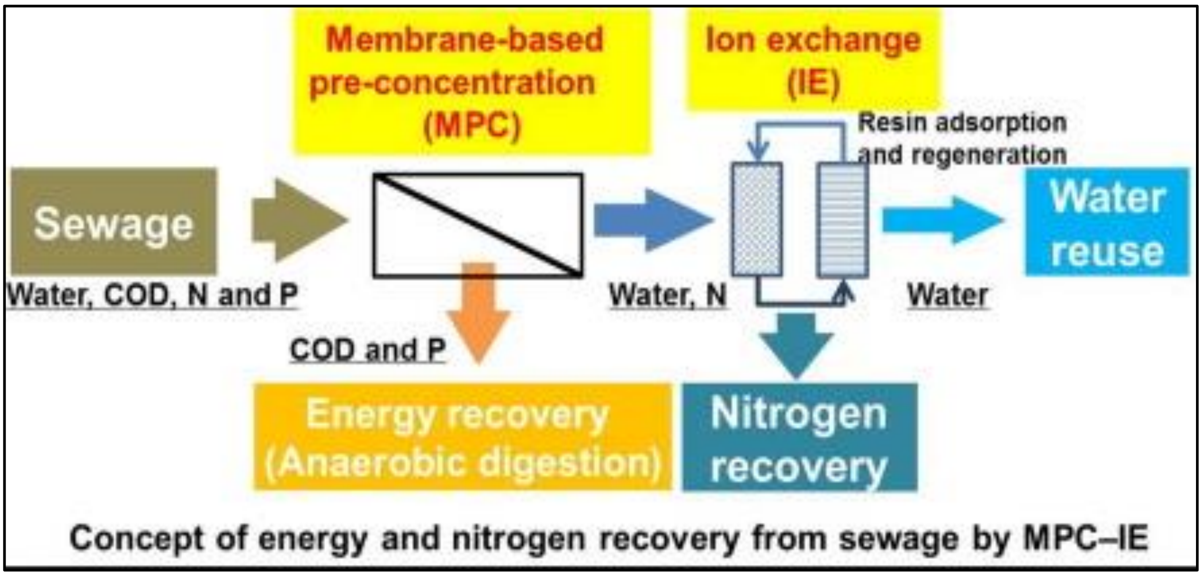


Figure 6: Membrane based pre-concentration coupled with IE (Gong et al., 2017)

2.6.4. Combined Coagulation Microfiltration

An intermittent aeration in combined coagulation microfiltration (CCM) system, not only save coagulant consumption required for scouring during a membrane fouling control but also conserve more organic matter and energy (Jin et al., 2016). The integration of the CCM and AD processes could achieve a net energy production of 0.0098 kWh/m^3 after deduction of 0.0919 kWh/m^3 required for the operation of the CCM system, thus showing promise as an effective organic matter concentration method for energy recovery from sewage. Compared to two typical technologies for sewage pre-concentration, i.e. direct sewage microfiltration (DSM) and continuous aerated sewage microfiltration (ASM), the CCM system under optimal aeration strategy showed higher concentration efficiency and slower permeability decline (i.e. better control of membrane fouling), and easier collection of retained organic matter (OM). A CCM reactor recovered 70% of an influent OM which was higher than the concentrate produced by a high-loaded membrane bioreactor (HL-MBR) at solids retention time (SRT) of one day. This explains recent attention in sewage treatment to minimizing energy consumption or even achieving net energy production, recovering the nutrients like phosphorus and nitrogen and reclaiming treated water. Among many choices, like microbial fuel cells (MFCs) which is capable to convert organic chemical energy of wastewater directly into energy (electricity or hydrogen) (Du et al., 2007), methane production by anaerobic digestion (AD) is regarded as a more mature technology for sewage energy recovery (Cao and Pawłowski, 2012). Accordingly, CCM is a relatively promising way of sewage pre-concentration compared to DSM and ASM (Jin et al., 2015). **Figure** 7 shows a typical setup of CCM process for the treatment of wastewater.

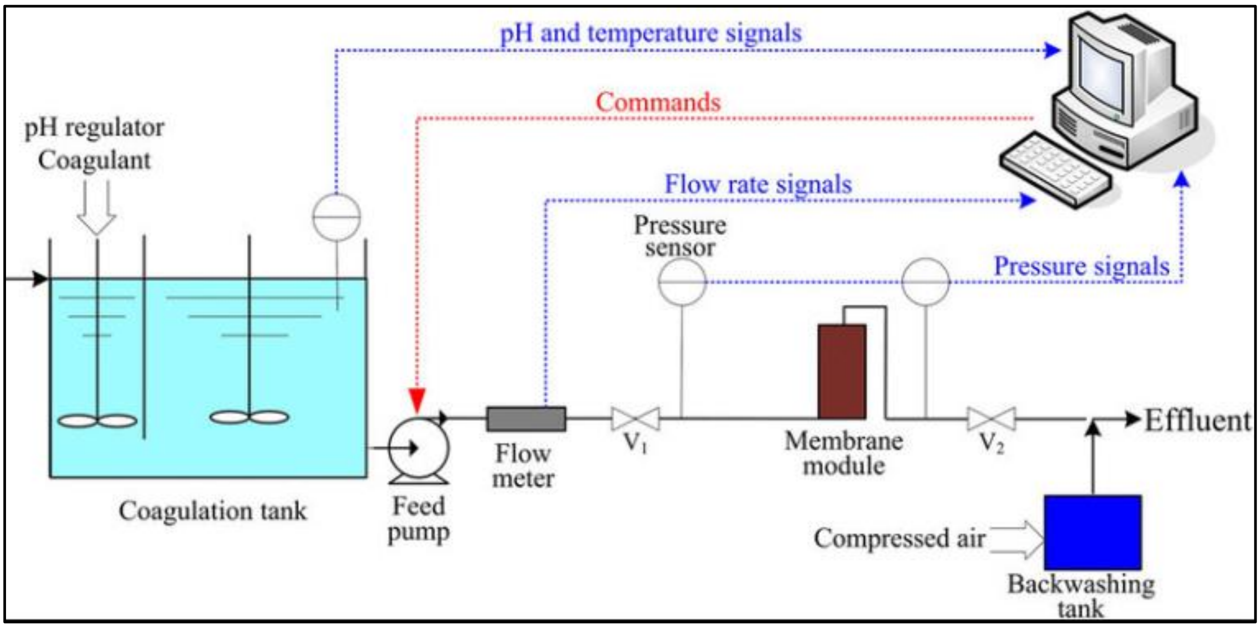


Figure 7: Coagulation Microfiltration (CMF) process (Jin et al., 2015)

2.6.5. Hybrid Coagulation Microfiltration

The idea of sewage concentration is gradually being accepted as a promising and sustainable way of wastewater resource recovery. In comparison to direct sewage microfiltration, (Jin et al., 2015) reported better filtration performance, concentration efficiency and decreased membrane fouling trends in Hybrid coagulation microfiltration (HCM) with air back flushing (ABF). HCM with AB recovered 70% of total influent organic matter with the COD concentration over 15,000 mg/L. HCM with ABF could be a promising effective sewage organic matter concentration for resource recovery under optimal conditions. Huge energy requirement in membrane based technologies and CO_2 emission (greenhouse gas) mainly due to aeration make aerobic technologies imperfect under the circumstances of global noticeable fossil fuel energy crisis and drastic climate change nowadays (McCarty et al., 2011). Anaerobic treatment has been demonstrated to be a mature and practical way of achieving net energy production and meeting the stringent effluent standards as well (Verstraete et al., 2009; Verstraete and Vlaeminck, 2011). However, direct anaerobic sewage treatment is often regarded as an uneconomical process considering the low organic loading in raw sewage.

Therefore, an appropriate pre-concentration step for organic matter is needed. This study investigated the performance of HCM with ABF on sewage organic matter concentration for resource recovery. The addition of coagulation process could mitigate the fouling trends and enhance the concentration efficiency in direct sewage micro-filtration (Mezohegyi et al., 2012).

Energy requirement in membrane based technologies and CO_2 emission (greenhouse gas) mainly due to aeration make aerobic technologies imperfect under the circumstances of global noticeable fossil fuel energy crisis and drastic climate change nowadays (McCarty et al., 2011). Therefore, anaerobic treatment of sewage has been demonstrated to be a mature and practical way of achieving net energy production and meeting the stringent effluent standards (Verstraete et al., 2009; Verstraete and Vlaeminck, 2011). Relatively high energy required for aerobic process and yields in more sludge production which required handling, treatment and finally disposal (Faust et al., 2014; Leitao et al., 2006; Shekdar, 2009). Therefore, anaerobic system was designed to get rid of problems mentioned above with aerobic process and to get up-concentrated effluent stream to produce maximum biogas at an infinite SRT. **Figure** 8 shows a layout of HCMF process for treating wastewater.

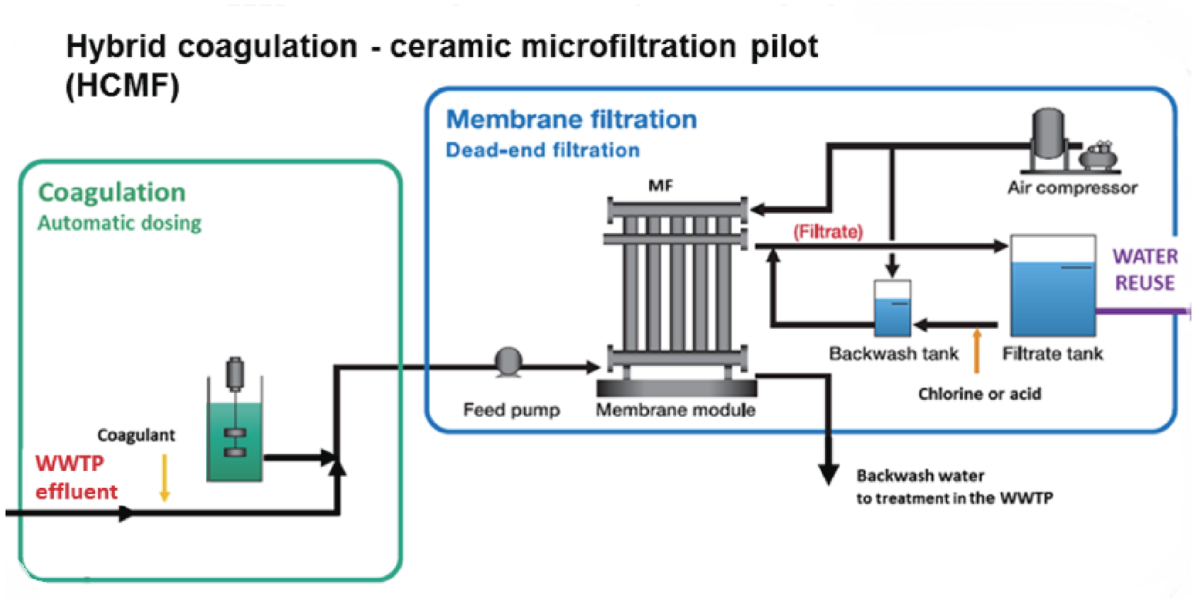


Figure 8: Layout of Hybrid Coagulation Microfiltration (HCMF) process (Faust et al., 2014)

2.6.6. Biosorption Sedimentation process

In literature, excess sludge stream has been reported as an up-concentrated stream or pre-concentrated stream for anaerobic digesters but not the effluent coming out of the system. The aim of this study was up-concentration of domestic wastewater in terms of COD and Total Phosphorous (TP) through Biosorption Sedimentation Process. Settled sludge of the anaerobic sedimentation tank was used as an adsorbent for up concentrating COD, TP and minimizing the ammonium concentration in concentrated stream. Finally, the optimized runs were obtained using Response Surface Methodology (RSM), keeping in view the Carbon: Nitrogen: Phosphorous (C: N: P) ratio required for biogas production via anaerobic digestion. The fact that sewage treatment still has to start from scratch in cities of the future, and also in many developing countries, offers an exquisite opportunity to directly choose a more sustainable approach to make treatment plants as net energy producers.

Chapter 3

Materials and Methods

3.1 System Design

System for up-concentration of domestic wastewater through Biosorption-Sedimentation (BS) process consists of feed tank, mixing tank of 2 Liters capacity and sedimentation tank with a capacity of 8 Liters (all tanks were made of acrylic sheet) as shown in **Figure** 9. Peristaltic pump (BT 300-2J, Longer, China) was used as a feed pump to control the flow rate of synthetic domestic wastewater into the mixing tank and other peristaltic pump (BT 300-2J, Longer, China) was used for sludge recirculation for maintaining mixed liquor suspended solids (MLSS) in a range of 0.8-1.2 g/L in mixing tank.

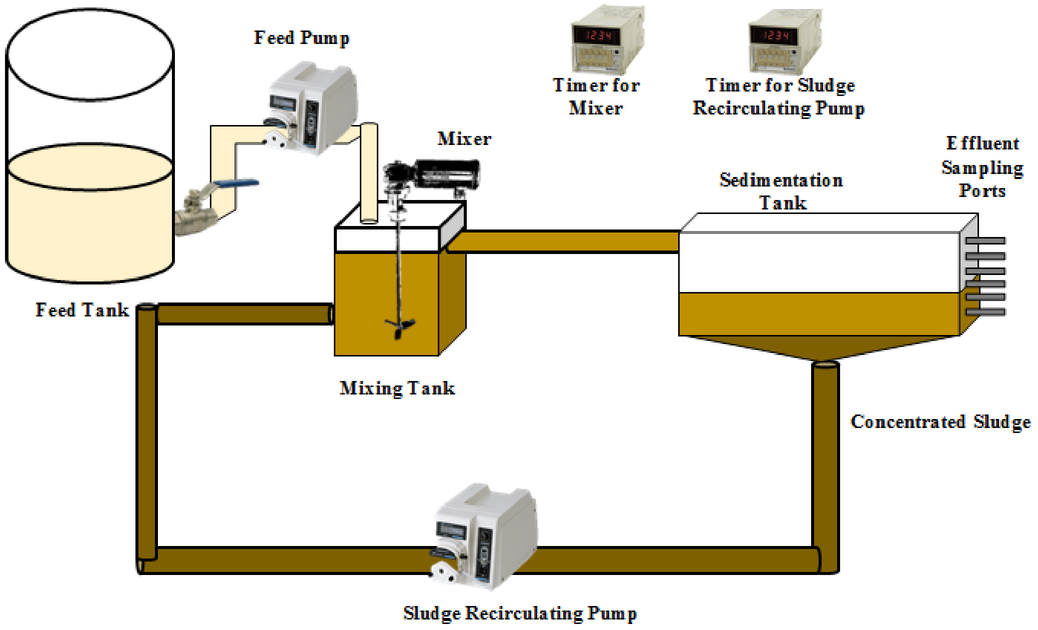


Figure 9: Process flow diagram of the Up-concentration system

In the mixing tank a propeller was used for the mixing of feed and sludge recirculation at a fixed 400 revolutions per minute (rpm), so that organic matter adsorb on the surface of microbes within the tank. Two Digital Display Time Relays (DH48S-S, Omron, Japan) were also used to ensure the proper working of the system. One time relay was connected with a propeller so that it moves for some time and then relaxed for a while and second time relays was linked with recirculate sludge pump to ensure the required MLSS concentration in mixing tank.

3.2 System Startup

Initially the system was operated in the batch mode for 15 days to find recirculation flow rate of sludge to maintain the required MLSS concentration within the mixing tank. A spike of nitrate (KNO_3) of 10 mg/l NO_3^{-1}–N was added to the initial anoxic phase to ensure that there was no limitation of electron acceptors for a complete nitrification & phosphorus uptake in mixing tank.

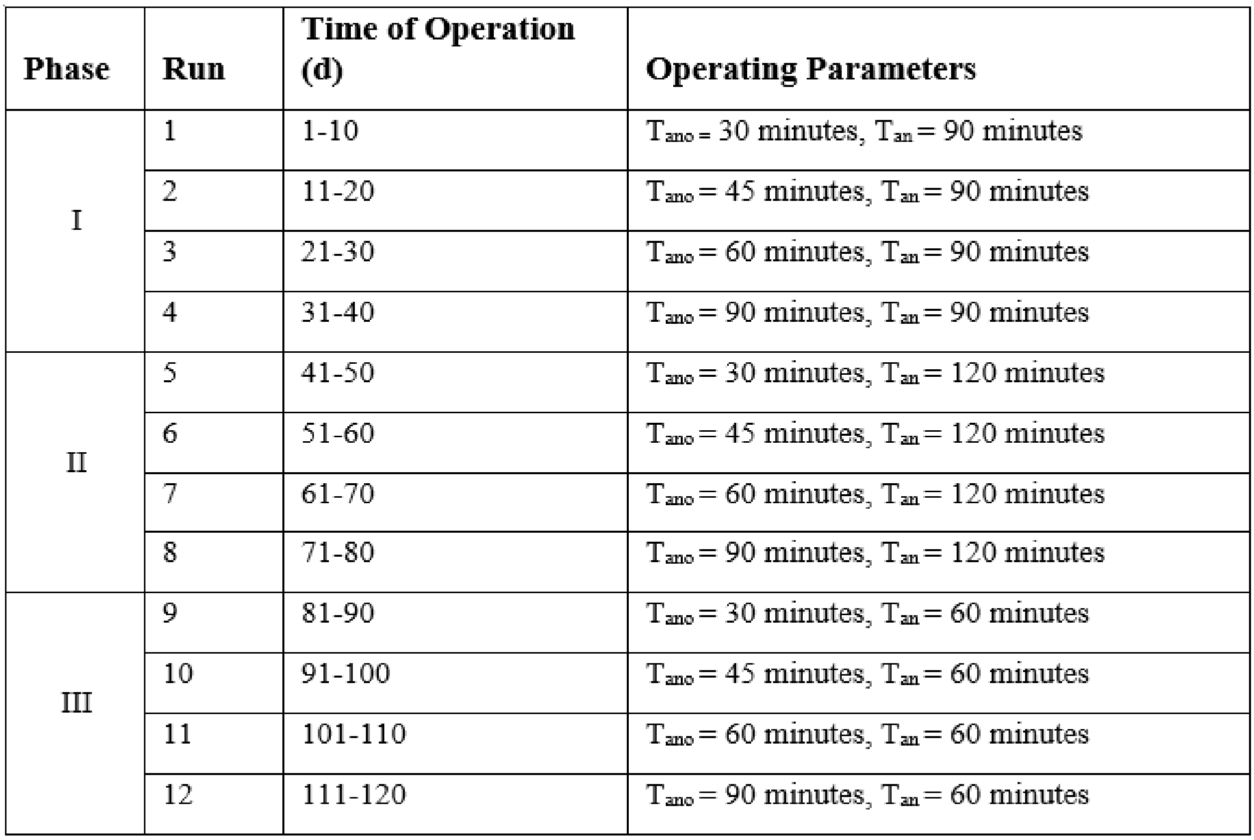
For startup, an acclimatized sludge was taken initially from Water and Wastewater Laboratory at Institute of Environmental Sciences & Engineering (IESE, NUST) and fed to sedimentation tank. Nitrogen purging was done for 10 minutes in order to ensure anaerobic condition within the sedimentation tank and for a few minutes within the mixing tank for making anoxic environment (Dissolved Oxygen must be less than 1.0 mg/L). Synthetic domestic wastewater of medium strength i.e. COD of 500 mg/L was used during the study and recipe of wastewater is reported in Table 3 (under sub-section 3.4). Recirculate pump was used to move anaerobic sludge from sedimentation tank to a mixing tank, where feed was introduced at a fixed flow rate through feed pump. Shorter Hydraulic Retention Time (HRT) of 30,45, 60 and 90 minutes was provided in mixing tank, so that biosorption occur. Micro-organisms within the mixing tank adsorbed organic matter present in wastewater and allowed to move in sedimentation tank, where minimum 60 minutes was given so that micro-organisms settle at the bottom and with up-concentrated effluent stream in terms of Chemical Oxygen Demand (COD) and Total Phosphorous (TP).

3.3 Research Design

Synthetic wastewater was introduced into the mixing tank through feed pump at constant flow rate in order to maintain HRT of the system and an acclimatized anaerobic sludge with mixed liquor volatile suspended solids (MLVSS) of 11 g/L was introduced into the mixing tank via peristaltic pump for finding an optimum recirculate rate at different HRTs of the system.

System was operated at different phases in order to find optimal HRT of anoxic tank and anaerobic tank for maximum biosorption of organic matter, total phosphorus but less concentration of ammonium-nitrogen in effluent. The operational phases of the system are shown in Table 1.

Table 1: Experimental Approach with operating parameters



Phase	**Run**	**Time of Operation (d)**	**Operating Parameters**
I	1	1-10	$T_{ano} = 30$ minutes, $T_{an} = 90$ minutes
	2	11-20	$T_{ano} = 45$ minutes, $T_{an} = 90$ minutes
	3	21-30	$T_{ano} = 60$ minutes, $T_{an} = 90$ minutes
	4	31-40	$T_{ano} = 90$ minutes, $T_{an} = 90$ minutes
II	5	41-50	$T_{ano} = 30$ minutes, $T_{an} = 120$ minutes
	6	51-60	$T_{ano} = 45$ minutes, $T_{an} = 120$ minutes
	7	61-70	$T_{ano} = 60$ minutes, $T_{an} = 120$ minutes
	8	71-80	$T_{ano} = 90$ minutes, $T_{an} = 120$ minutes
III	9	81-90	$T_{ano} = 30$ minutes, $T_{an} = 60$ minutes
	10	91-100	$T_{ano} = 45$ minutes, $T_{an} = 60$ minutes
	11	101-110	$T_{ano} = 60$ minutes, $T_{an} = 60$ minutes
	12	111-120	$T_{ano} = 90$ minutes, $T_{an} = 60$ minutes

The temperature within reactor was in the range of 28-32^{o}C as per ambient temperature. Table 2 presents the operating conditions of the system.

Table 2: Operating conditions of reactors

Reactor	Variable Parameters	Operation Description
Mixing Tank	Anoxic phase with T_{ano} = 30, 45, 60 & 90 minutes	Recirculate Sludge from anaerobic sedimentation tank to mixing tank operated at different HRTs in order to find how the values of COD, TP, Ammonium-N and TKN varies with HRT.
Sedimentation Tank	Anaerobic phase with T_{an} = 60, 90 & 120 minutes	Volume of sludge directly varies to HRT and recirculate ratio was found for maintaining MLSS in the range of 850-1200 mg/L in a mixing tank for each phase.

Experiments were conducted to find the concentrations of chemical oxygen demand (COD) total phosphorous (TP), ammonium-nitrogen (NH_4^+-N) and total kjeldahl nitrogen (TKN) in effluent against different anoxic and anaerobic HRTs.

3.4 Domestic Wastewater Characteristics

The domestic wastewater recipe used for this study is presented in Table 3. The average concentrations of COD, NH_4^+ –N, total kjeldahl nitrogen (TKN) and total phosphorus (TP) in the synthetic wastewater over the experimental period were 500 ± 15, 90 ± 10, 45 ± 8 and 6 ± 1.5 mg/l, respectively. The pH of the influent varied between 6.81 and 7.88 with the mean value of 7.20. Table 3 shows the ingredients of synthetic domestic wastewater along with concentrations in mg/L.

Table 3: Synthetic Domestic Wastewater

Sr.#	Chemicals	Weight (g)
1.	Glucose ($C_6H_{12}O_6$)	90
2.	Ammonium Chloride (NH_4Cl)	34.38
3.	Potassium dihydrogen Phosphate (KH_2PO_4)	4.29
4.	Sodium Bicarbonate ($NaHCO_3$)	18

Sr.#	Chemicals	Weight (g)
5.	Calcium Chloride ($CaCl_2$)	0.875
6.	Magnesium Sulphate ($MgSO_4.7H_2O$)	0.875
7.	Ferric Chloride ($FeCl_3$)	0.09
8.	Cobalt Chloride ($CoCl_2$)	0.018
9.	Zinc Chloride ($ZnCl_2$)	0.018
10.	Nickle Chloride (NiCl)	0.018

3.5 Analytical Methods

3.5.1. Treatment Performances

Treatment performance of the effluent was checked by performing the following tests by adopting Standards Methods (APHA et al.,2012).

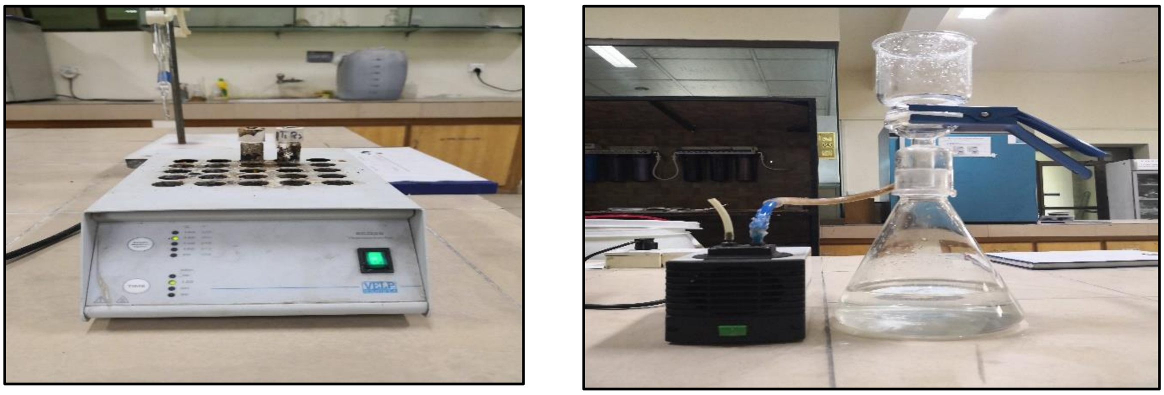
- Chemical Oxygen Demand (COD)
- Total Phosphorous (TP)
- Ammonium-Nitrogen (NH_4^+-N)
- Total Kjheldal Nitrogen (TKN)
- Alkalinity and Volatile Fatty Acids (VFAs)

3.5.2. Biomass Characteristics

Following biomass characteristics were performed throughout the experimental period by adopting Filtration Evaporation Method.

- Mixed Liquor Suspended Solids (MLSS)
- Mixed Liquor Volatile Suspended Solids (MLVSS)

Pictures of the equipment's that were used during the study are shown in **Figure** 10.



COD Analyzer

Filtration Assembly

Oven

Muffle Furnace

Figure 10: Pictorial View of Equipment's

3.5.3 Optimization of the process by using Response Surface Methodology (RSM)

RSM was not only applied to get various conditions of the studied factors, but also used in order to obtain optimized run of the study. As, RSM is a statistical tool used to analyze, optimize and evaluate the process parameters. It defines the effect of the independent factors alone or in combination and also generates a mathematical model which describes the processes interaction (Kumar et al., 2008). In this study, process optimization was performed influenced by independent factors i.e. anoxic and anaerobic HRT, using Box Behnken Design

(BBD). It was also used to find relationship between factors (anoxic and anaerobic HRTs) and responses (COD, TP, Ammonium-Nitrogen and TKN).

Chapter 4

Results and Discussion

4.1 Effect of Anoxic HRT on Concentration Factor for COD

The experiments were conducted at anoxic HRT of 30, 45, 60 and 90 minutes against anaerobic HRT of 60, 90 and 120 minutes as presented in Table 1. Up-concentration of COD was observed to increase sharply with an increase in anoxic HRT up to 60 minutes and then decrease at 90 minutes by keeping anaerobic HRT of 60 minutes as constant. The same trends were observed at 90 and 120 minutes of anaerobic HRT as shown in Fig. 11. Up to 8 times up-concentration of COD in effluent was observed at T_{ano}/T_{an} of 60 minutes/60 minutes due to more VSS concentration and VSS/TSS ratio as discussed under sub-section 3.5.

Physical processes like adsorption and bio-flocculation are endorsed to shorter HRTs (Leal et al., 2010). Due to the presence of more active bacteria at shorter anoxic HRT and more VSS/TSS ratio at lower anaerobic HRT, results in more adsorption of organic matter on microbial surface consequently more COD in effluent. The extracellular polymeric substances (EPS) might acts as a binding material for biosorption of organic matter. Apart from sorption of organic compounds, EPS has multiple functions include, flocs formation due to aggregation of bacterial cells and floc structure stabilization (Comte et al., 2006; Ledin, 2000).

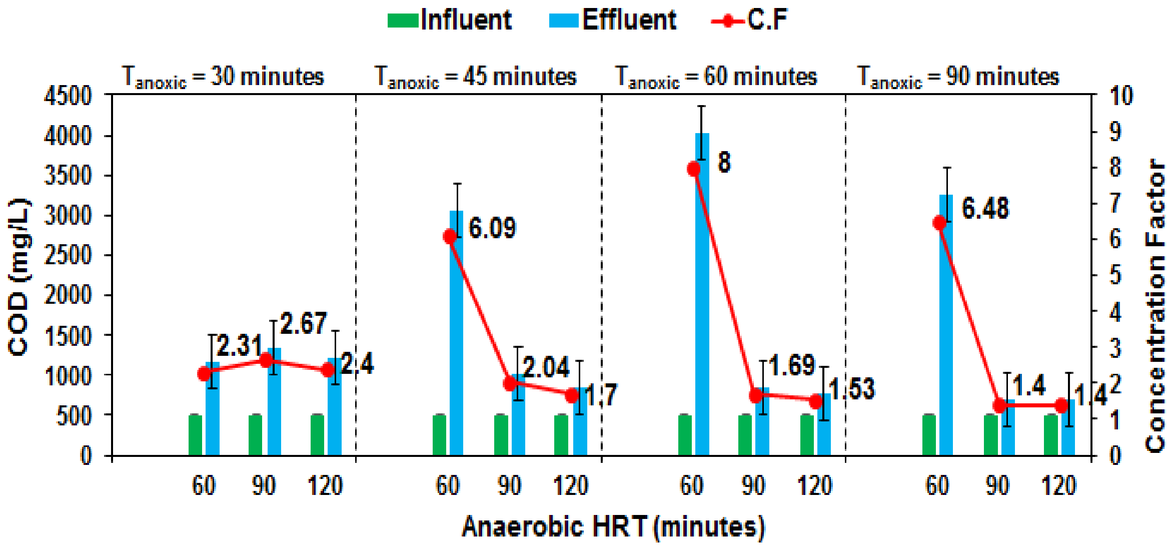


Figure 11: COD up-concentration against anaerobic HRT

4.2 Total Phosphorous in Effluent against Anaerobic HRTs

With an increase in anoxic HRTs from (30 minutes to 90 minutes), concentration factor of total phosphorus decrease from 1.96 to 1.84 for an anaerobic HRT (60 minutes) as shown in Fig. 12 . Similar trend was also exhibited by other anaerobic HRTs of 90 and 120 minutes. Very minute decrease in phosphorous concentration occurs due to increase in anoxic HRT (Wang et al., 2009). With respect to an increase in anoxic HRT, the potential for anoxic phosphorous uptake increases by Phosphorous Accumulating Organisms (PAOs), which are less efficient than aerobic phosphorous uptake (Hu et al., 2002). While anaerobic HRT influenced more on phosphates release in anaerobic reactor of the system. Significantly, decrease in phosphate concentration was observed with an increasing anaerobic HRT during the study.

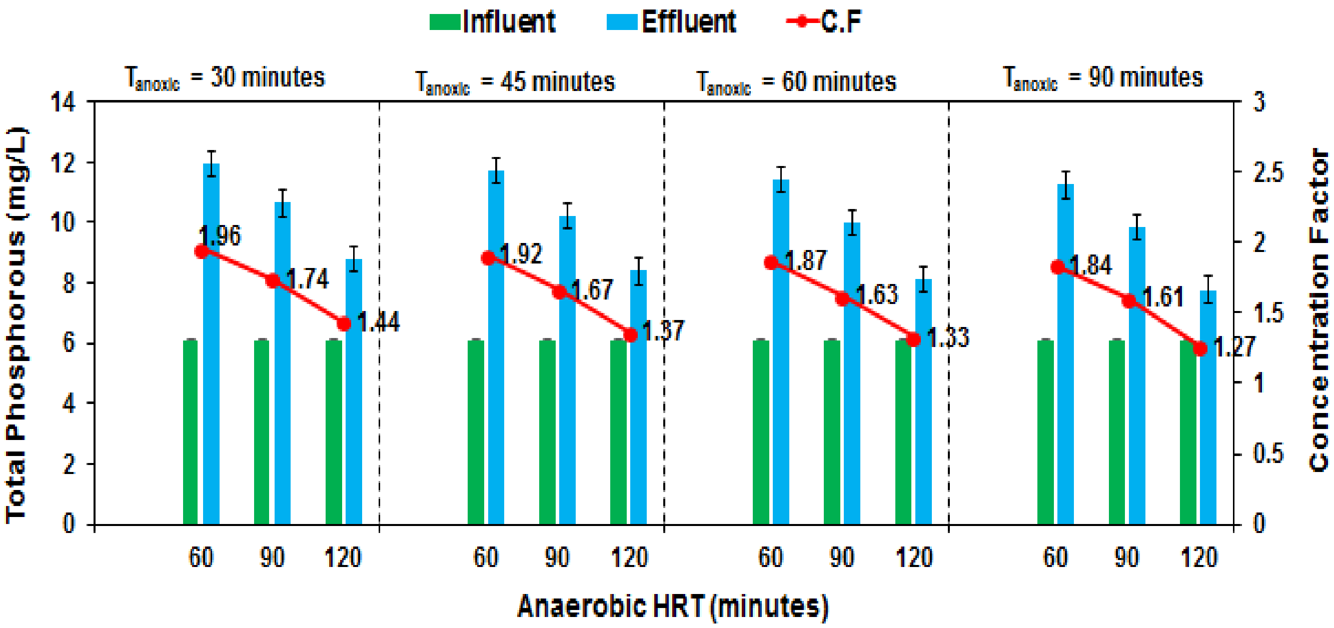


Figure 12: Total Phosphorous (TP) up-concentration against anaerobic HRT

4.3 Ammonium-Nitrogen Removal at Varying HRTs

Nitrification is a two-step process in which ammonium nitrogen is converted into nitrites followed by nitrates in the presence of oxygen, which acts as an electron acceptor. In this study, nitrification was achieved under anoxic condition (mixing tank of the system) and soluble COD reported as an electron acceptor instead of oxygen (Kim et al., 2004). Removal efficiency of ammonium-nitrogen declined significantly by changing anoxic HRT from 30 to 90 minutes (at constant anaerobic HRT) as shown in Fig. 13. Maximum removal efficiency of 93% was achieved at T_{ano}/T_{an} of 30 minutes/90 minutes. Removal of ammonium nitrogen under anoxic/anaerobic condition occurred which might be due to nitrites production by the process termed as Nitritation in a mixing tank (Fux et al., 2002) and anaerobic ammonium oxidation (ANAMOX) process. Nitrites have been reported as an electron acceptor instead of oxygen mandatory for the oxidation of ammonium-nitrogen (Karthikeyan and Joseph, 2007). Furthermore, higher F/M ratio (shorter anoxic HRT) led to enhanced biomass yield and activity which consequently improved removal efficiency of nitrogen (Song et al., 2008).

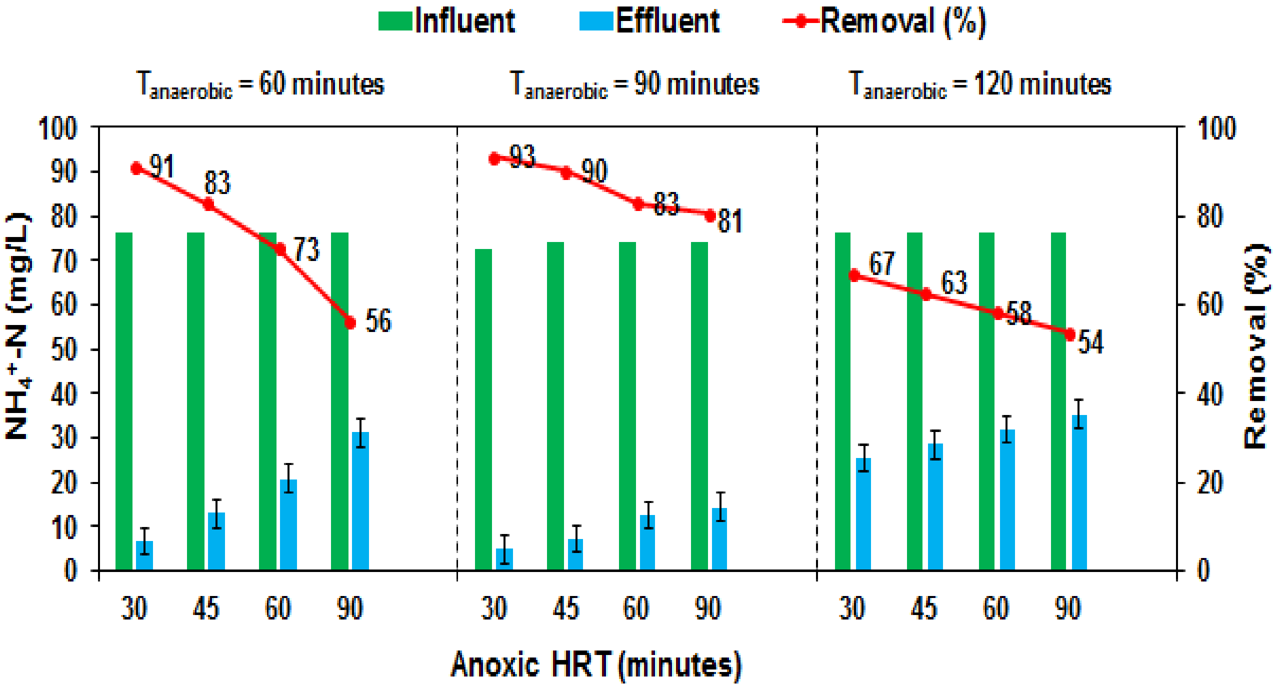


Figure. 13. Removal Percentages of Ammonium-Nitrogen at Different Anoxic HRTs

4.4 TKN Removal at various Anaerobic HRTs

TKN removal percentage decreases significantly with an increase in anoxic HRT (from 30 minutes to 90 minutes) at constant anaerobic HRT of 60, 90 and 120 minutes, as illustrated in **Figure**. 14. Removal efficiency of nitrogen tends to decrease with an increase in HRT (Cho et al., 2005), which was also observed in this study. Shorter HRT resulting in an enhanced biological capacity, activity of denitrifying bacteria and more F/M ratio (Song et al., 2009). A high SRT is correlated with a greater microbial diversity due to a low selective pressure on the present microbial community. Because of this, the slower growing nitrifying and denitrifying organisms are able to grow which makes nitrogen removal possible (Ekama, 2010).

4.5 VSS to TSS ratio of Recycled Sludge

VSS concentration increased with an increase in T_{ano}/T_{an} ratio (particularly at anaerobic HRT of 90 and 120 minutes). While at anaerobic HRT of 60 minutes, VSS concentration found to increase initially and then slightly decrease with an increase in anoxic HRT, as shown in **Figure.** 15. VSS/TSS ratio found to decrease with an increase in anaerobic HRT (at constant anoxic HRT) except for 30/60 ratio (T_{ano}/T_{an}). As higher HRT yield lower F/M ratio, resulting in more time for microbes to consume organic matter and ultimately entering in their death/endogenous phase (Diez et al., 2002). Furthermore, MLSS concentration and the sludge viscosity varies indirectly with the system's HRT (Meng et al., 2007).

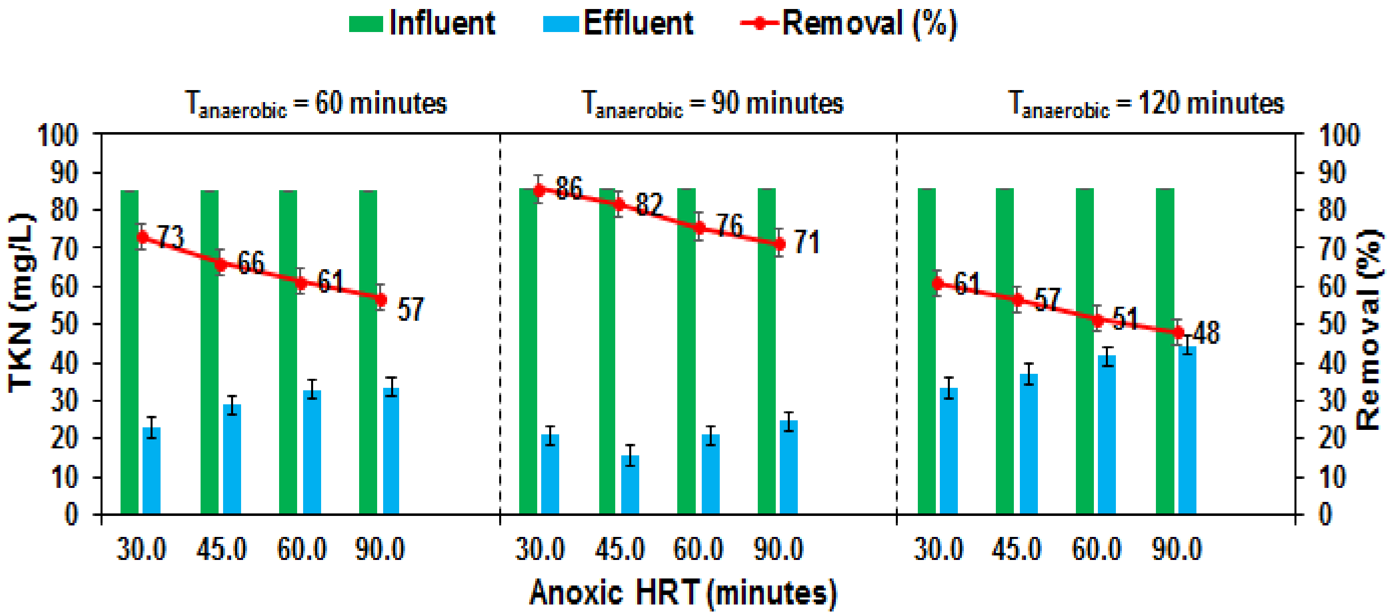


Figure 14 : TKN removal against anaerobic HRT

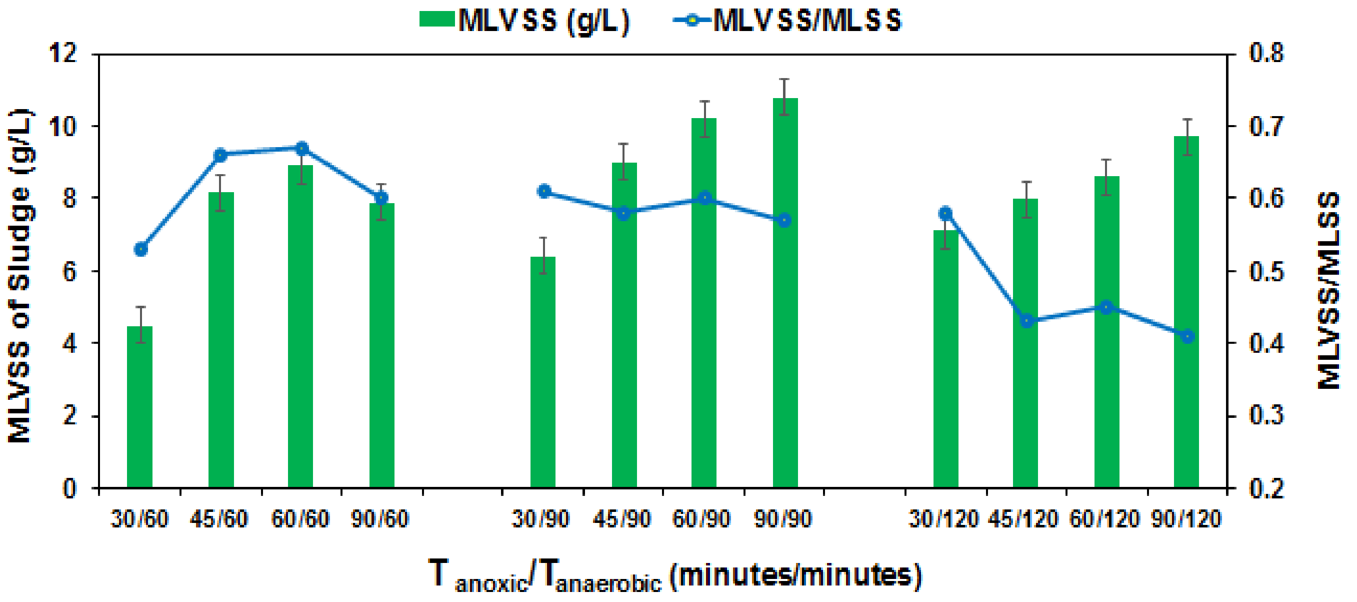


Figure 15: VSS/TSS ratio of Anaerobic Sludge at Different HRTs

4.6 Ammonium-Nitrogen and TKN Removal against Different F/M Ratios

Ammonium-nitrogen and TKN removal efficiency increases as anaerobic HRT of sedimentation tank decreases as shown in **Figure**. 16. More volatile suspended solids (VSS) concentration was found at shorter anaerobic HRT. The reason for decrease in removal efficiency may be due to the fact of less contact time available for microbes at shorter HRT within the sedimentation tank for completion of denitrification process. Optimum anoxic/anaerobic HRT was found to be 30 minutes/60 minutes for maximum removal of ammonium-nitrogen and total khjeldal nitrogen from the system. As higher HRT results in lower F/M ratio and ends up with decrease removal efficiency of nitrogen (Song et al., 2009).

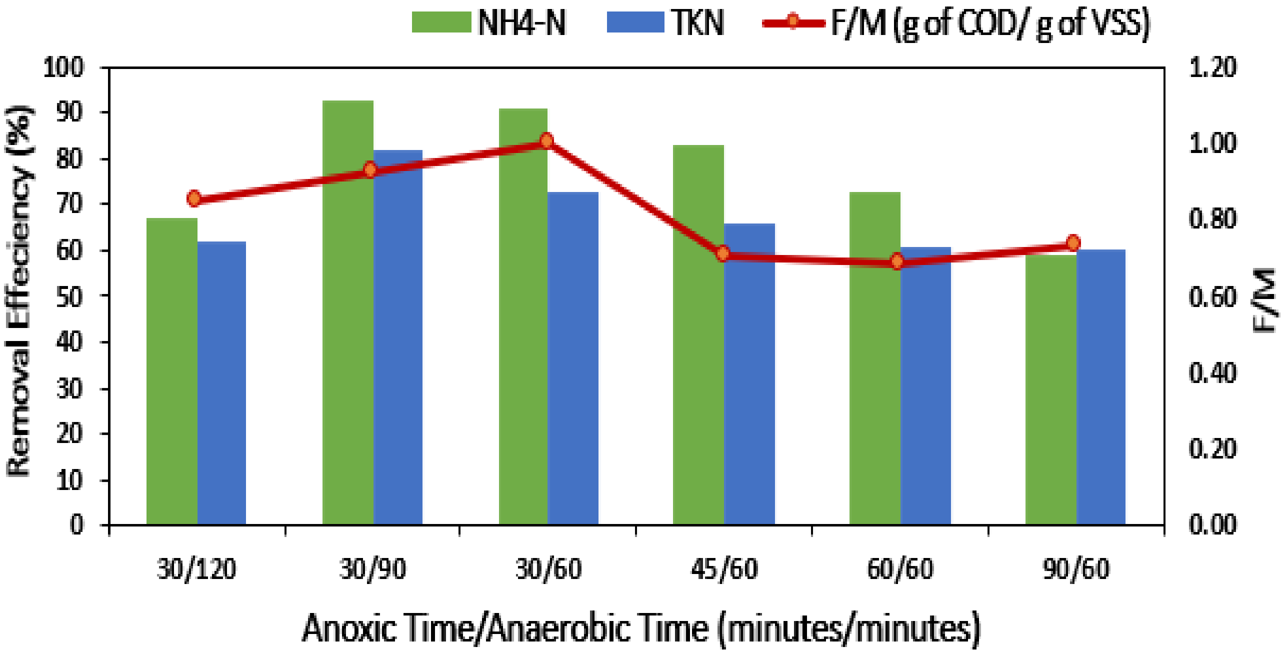


Figure 16: Removal Efficiencies of NH_4-N & TKN against F/M ratio

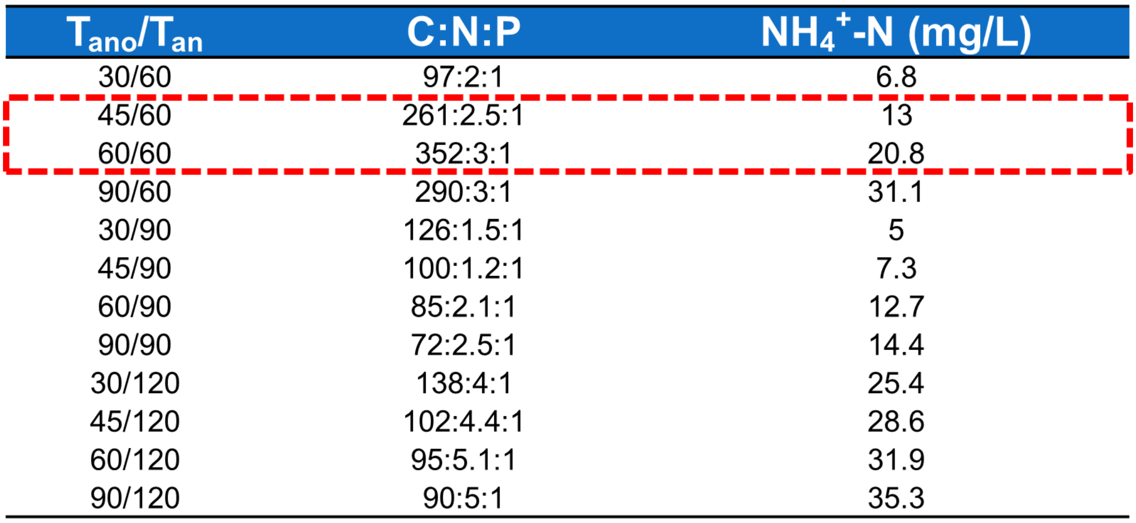
The removal efficiency of ammonium-nitrogen in particular with anoxic HRT (constant anaerobic HRT) and F/M is shown in **Figure**. 16. Shorter anoxic HRT cedes more nitrates, final product of nitrification process. Furthermore, inducing higher F/M ratio which lead to enhanced biomass yield and biomass activity, consequently improved removal efficiency of nitrogen (Song et al., 2008).

4.7 Selection of Optimized Run

4.7.1 According to CNP ratio

Based on carbon to nitrogen ratio (C:N), nitrogen to phosphorous ratio (N:P) and concentration of ammonium-nitrogen in effluent that was supposed to be the feed of anaerobic digestion for biogas production. T_{ano}/T_{an} of 45/60 and 60/60 were selected as optimized runs having carbon to phosphorous ratio (C:P) of 261 and 352, COD to Nitrogen ratio (COD:N) of 104 and 117, respectively. Up to 15 times decrease in phosphorous concentration against COD did not significantly affect the efficiency of the reactor in terms of biogas production (Britz et al. (1988). Biogas production ceased at COD to Nitrogen ratio of 750 and maximum COD removal from anaerobic digester was reported at COD:N of 250 (Hussain et al., 2015). Table 4 shows the C:N:P ratio of all runs at different anoxic and anaerobic HRTs.

Table 4: CNP ratio of different runs



T_{ano}/T_{an}	C:N:P	NH_4^+-N (mg/L)
30/60	97:2:1	6.8
45/60	261:2.5:1	13
60/60	352:3:1	20.8
90/60	290:3:1	31.1
30/90	126:1.5:1	5
45/90	100:1.2:1	7.3
60/90	85:2.1:1	12.7
90/90	72:2.5:1	14.4
30/120	138:4:1	25.4
45/120	102:4.4:1	28.6
60/120	95:5.1:1	31.9
90/120	90:5:1	35.3

4.7.2 *Maximum Potential Energy within up concentrated effluent*

Theoretically, maximum potential energy of effluent was calculated against different anoxic to anaerobic HRTs (Khiewwijit et al., 2015). Runs with T_{ano} /T_{an} (45/60 and 60/60) were selected as optimized runs with reference to maximum potential energy stored as total COD (TCOD) of the effluent as shown in Table 5.

Table 5: Maximum Potential of Biogas production in effluent stream at different T_{ano}/T_{an} Ratio

T_{ano}/T_{an}	30/60	45/60	60/60	90/60	30/90	45/90	60/90	90/90	30/120	45/120	60/120	90/120
TCOD	1162	3065	4024	3264	1344	1026	851	705	1209	855	771	703
bsCOD[1]	203.4	536.3	704.2	571.2	235.2	179.5	148.9	123.4	211.6	149.6	134.9	123.0
bpCOD	672.8	1774.6	2329.9	1889.9	778.2	594.1	492.7	408.2	700.0	495.1	446.4	407.0
nbsCOD	87.2	229.9	301.8	244.8	100.8	77.0	63.8	52.9	90.7	64.1	57.8	52.7
nbpCOD[2]	198.7	524.2	688.1	558.1	229.8	175.4	145.5	120.6	206.7	146.2	131.8	120.2
NH_4^+-N	6.8	13.0	20.8	31.1	5.0	7.3	12.7	14.4	25.4	28.6	31.9	35.3
TP	12.0	11.7	11.4	11.2	10.6	10.2	10.0	9.8	8.8	8.4	8.1	7.8
Max P.E (kWh/kg of COD)[3]	4.49	11.83	15.53	12.60	5.19	3.96	3.28	2.72	4.67	3.30	2.98	2.71

4.7.3 Optimized run predicted by RSM

Optimized runs were predicted by RSM, investigating COD, TP, ammonium-nitrogen and TKN as responses against the two factors (anoxic and anaerobic HRTs), on the basis of their probably interaction and importance by using Box Behnken Design. It suggested two optimum solutions as shown in Table 6.

Table 6: Optimal Solution Predicted by RSM Software

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[1] COD fractions determination based upon 17.5% for CODbs, 17.1% for CODnbp, 57.9% for CODpb, and 7.5% for CODnbs Pasztor, I., Thury, P., Pulai, J., 2009. Chemical oxygen demand fractions of municipal wastewater for modeling of wastewater treatment. International Journal of Environmental Science & Technology 6, 51-56..

[2] Non-biodegradable organic matter will contribute to total sludge production Metcalf, E., Eddy, H., 2003. Wastewater engineering: treatment and reuse. Wastewater Engineering, Treatment, Disposal and Reuse. Techobanoglous G, Burton FL, Stensel HD. Tata McGraw-Hill Publishing Company Limited, 4th edition. New Delhi, India..

[3] Theoretically, 3.86 kWh energy production per kg of COD oxidation to carbon dioxide and water Khiewwijit, R., Temmink, H., Rijnaarts, H., Keesman, K.J., 2015. Energy and nutrient recovery for municipal wastewater treatment: How to design a feasible plant layout? Environmental Modelling & Software 68, 156-165.

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Anaerobic HRT	is in range	60	120	1	1	5
Anoxic HRT	is in range	30	90	1	1	5
COD	maximize	703	4024	1	1	5
TP	maximize	7.78	11.97	1	1	5
NH4-N	minimize	5	35.3	1	1	5
TKN	maximize	12.3	44.5	1	1	5

Solutions

Number	Anaerobic HRT	Anoxic HRT	COD	TP	NH_4^+-N	TKN	Desirability
1	60	56	3608.19	11.45	19.13	34.04	0.72
2	111.11	30	1268.17	9.29	18.29	24.81	0.34

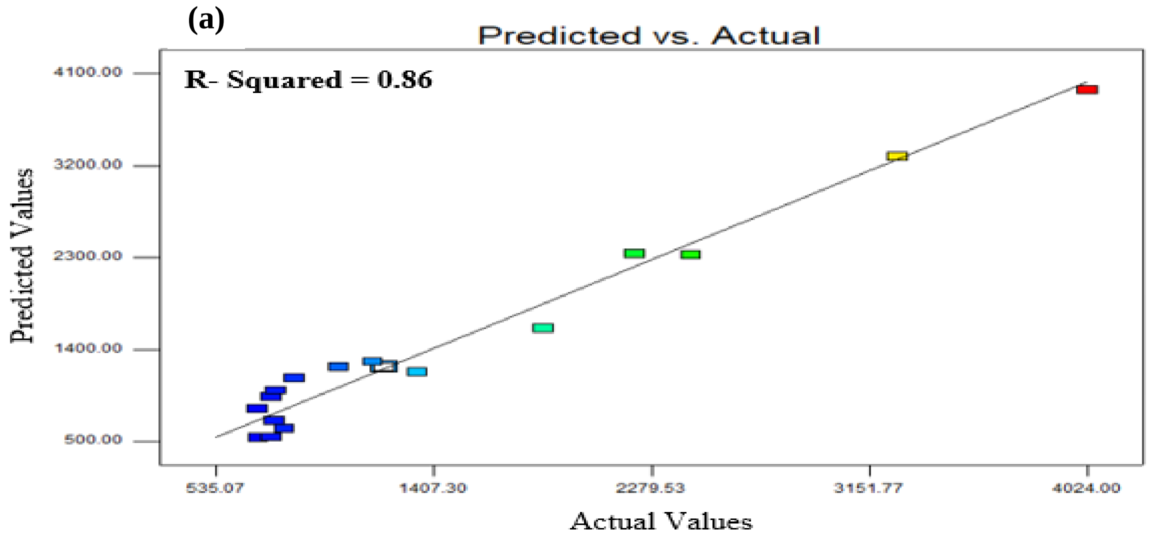
4.7.4 RSM response to COD, TP, NH_4^+-N and TKN

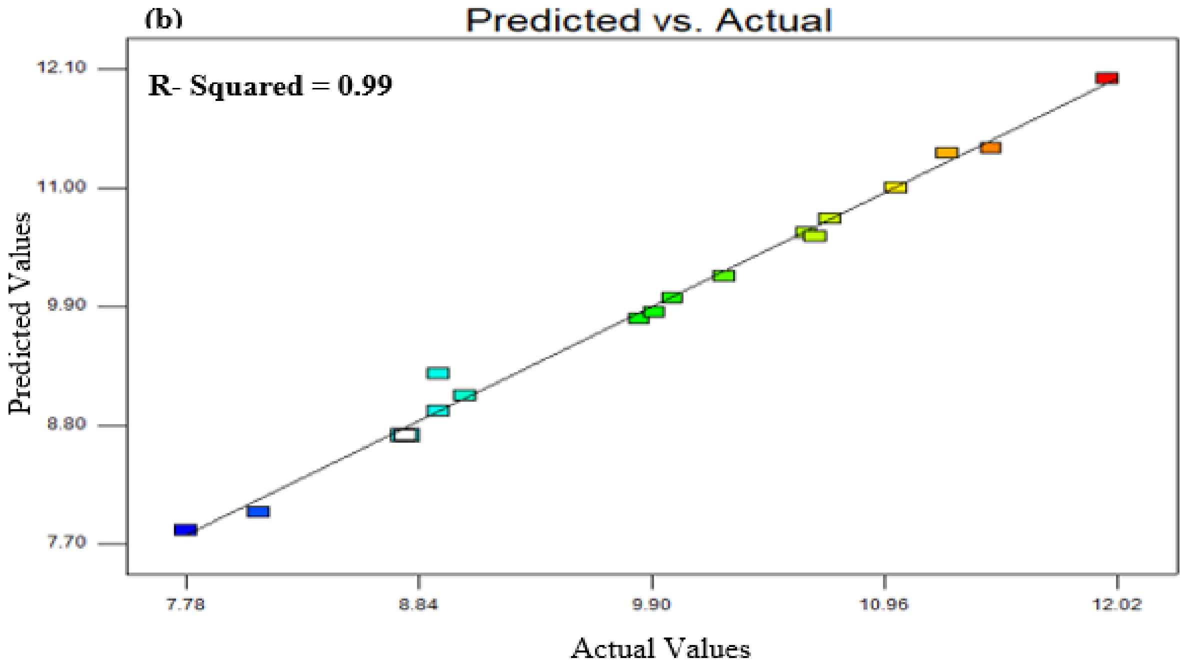
A statistical tool, analysis of variance (ANOVA) was used for analyzing the results and to get regression equations which fit the predicted and actual data. Regression co-efficient (R^2) value close to one, not only indicates the existence of a strong relationship between the variables but also authenticates the validity of the actual data.

Following regression equations presents dependence of every parameter (COD, TP, NH_4^+-N and TKN) on two factors i.e. A = Anaerobic HRT, B = Anoxic HRT and equations with R^2 values are given below:

- $COD = +1216.49 - 1076.13*A + 106.11*B - 686.95*A*B + 981.51*A^2 - 480.67*B^2$ ($R^2 = 0.86$)
- $TP = +9.95 - 1.69*A - 0.38*B - 0.033* A*B - 0.24*A^2 + 0.24*B^2$ ($R^2 = 0.99$)
- $NH_4^+\text{-}N = +14.88 + 5.85*A + 8.02*B - 4.07*A*B + 11.74*A^2 - 1.37*B^2$ ($R^2 = 0.93$)
- $TKN = +23.16 + 5.33*A + 3.78*B - 0.15*A* B + 16.85*A^2 - 6.77*B^2$ ($R^2 = 0.85$)

Regressions co-efficient (R^2) values indicating strong relation between actual values (experimental values) against the predicted values (predicted by RSM software) as shown in Fig. 18 (a, b, c and d).


(a)
Predicted vs. Actual
R- Squared = 0.86
Predicted Values
4100.00
3200.00
2300.00
1400.00
500.00
535.07
1407.30
2279.53
3151.77
4024.00
Actual Values


(b)
Predicted vs. Actual
R- Squared = 0.99
Predicted Values
12.10
11.00
9.90
8.80
7.70
7.78
8.84
9.90
10.96
12.02
Actual Values

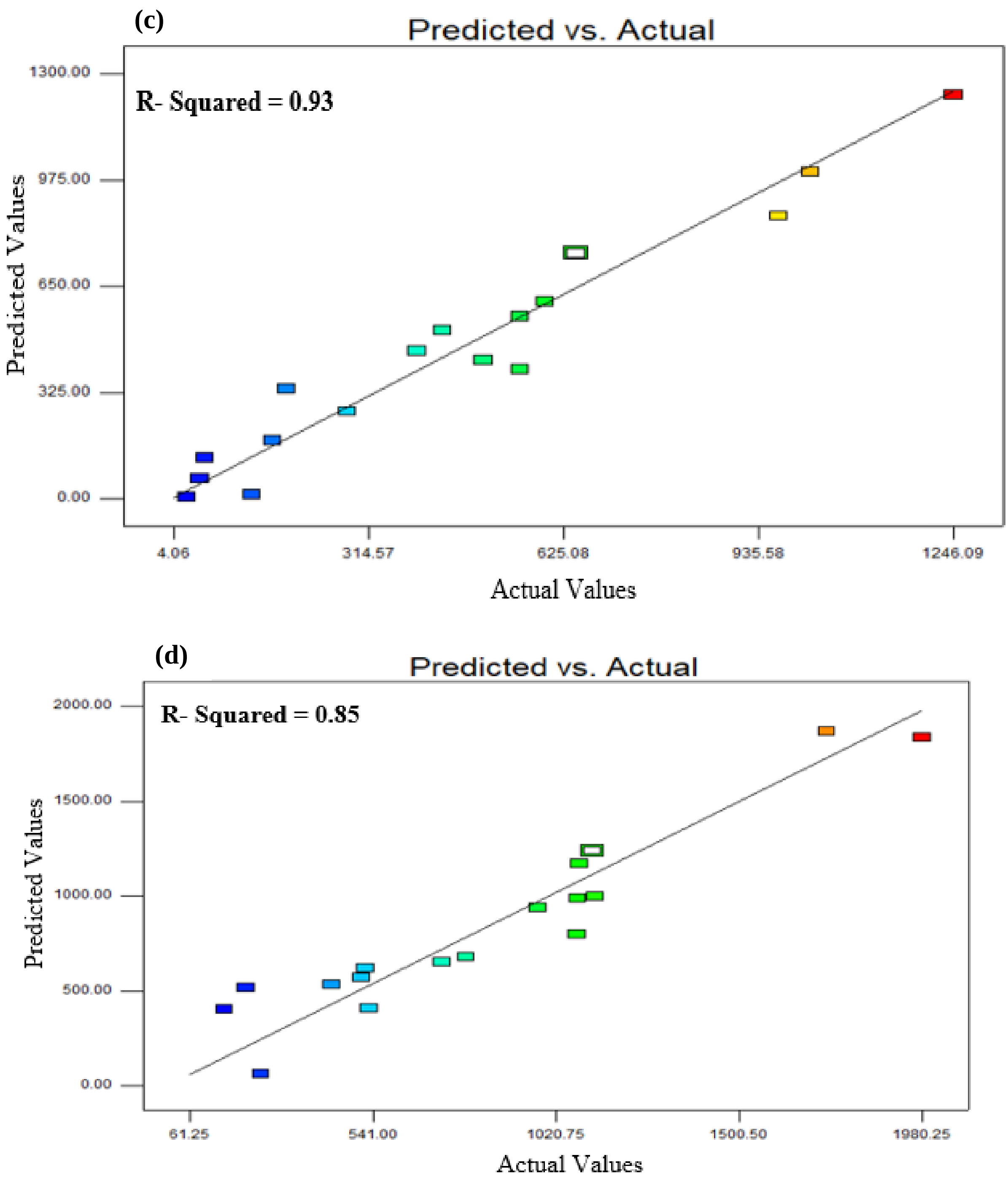
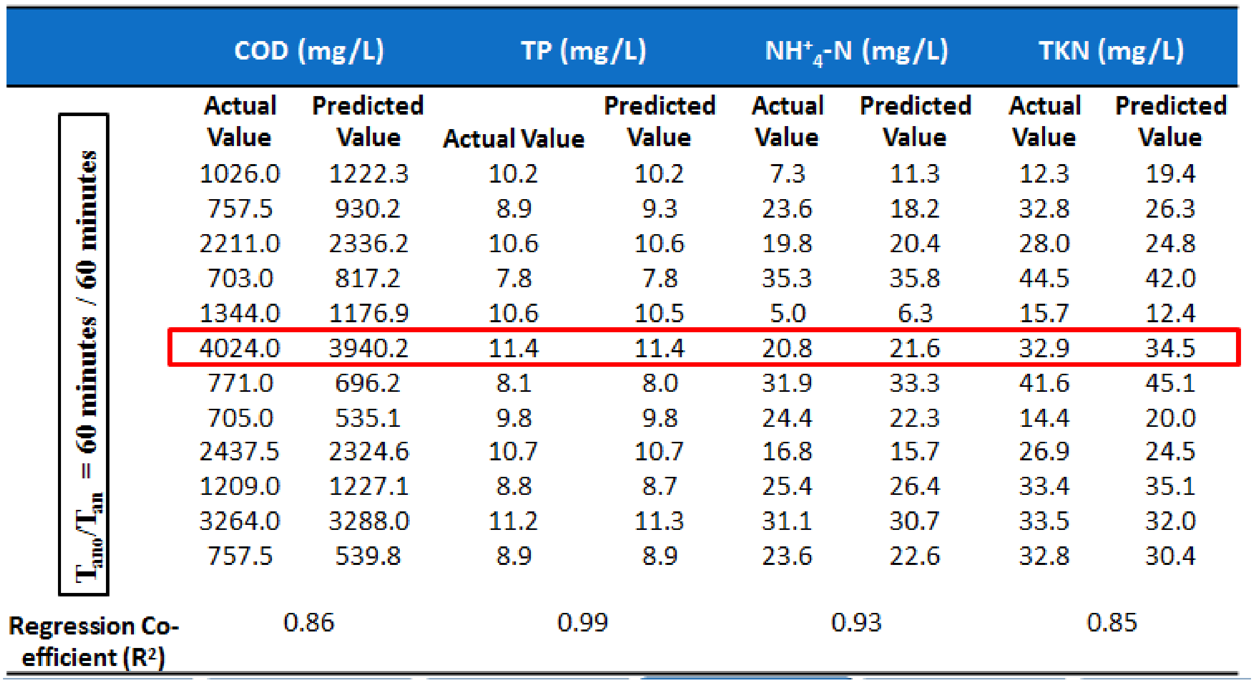


Figure 17: (a) COD (b) Total Phosphorous (TP) (c) Ammonium Nitrogen (d) TKN

Predicted verses actual values of all four responses of the study i.e. COD, TP, NH_4^+-N and TKN has been shown in Fig. 19 with the most COD value (predicted and actual) was found at a ratio of anoxic HRT to anaerobic HRT (T_{ano}:T_{an}) of 60 minutes to 60 minutes. Also, the optimum run predicted by RSM software was also repeated as a duplicate run with no significant difference (less than 6 %) found in values of all responses against anoxic HRT to anaerobic HRT ratio (T_{ano}:T_{an}).



	COD (mg/L)		TP (mg/L)		NH^+_4-N (mg/L)		TKN (mg/L)	
	Actual Value	Predicted Value	Actual Value	Predicted Value	Actual Value	Predicted Value	Actual Value	Predicted Value
T_{ano}/T_{an} = 60 minutes / 60 minutes	1026.0	1222.3	10.2	10.2	7.3	11.3	12.3	19.4
	757.5	930.2	8.9	9.3	23.6	18.2	32.8	26.3
	2211.0	2336.2	10.6	10.6	19.8	20.4	28.0	24.8
	703.0	817.2	7.8	7.8	35.3	35.8	44.5	42.0
	1344.0	1176.9	10.6	10.5	5.0	6.3	15.7	12.4
	4024.0	3940.2	11.4	11.4	20.8	21.6	32.9	34.5
	771.0	696.2	8.1	8.0	31.9	33.3	41.6	45.1
	705.0	535.1	9.8	9.8	24.4	22.3	14.4	20.0
	2437.5	2324.6	10.7	10.7	16.8	15.7	26.9	24.5
	1209.0	1227.1	8.8	8.7	25.4	26.4	33.4	35.1
	3264.0	3288.0	11.2	11.3	31.1	30.7	33.5	32.0
	757.5	539.8	8.9	8.9	23.6	22.6	32.8	30.4
Regression Co-efficient (R^2)	0.86		0.99		0.93		0.85	

Figure 18: Actual vs Predicted values along with regression co-efficient

4.7.5 Confirmation of optimized run suggested by RSM

Optimized run suggested by RSM was performed as a duplicate run in order to check how the values (actual versus predicted) varied and to confirm the authenticity of Box Behnken design of RSM. Table 7 shows the values of responses (actual and predicted) of an optimized run at 60 minutes/60 minutes (T_{ano}/T_{an}).

Table 7: Actual versus predicted values of an optimized run (Duplicate Run)

Factors	Experimental Values (mg/L)	Predicted Values by RSM (mg/L)
COD	4024.0	3940.2
TP	11.4	11.36
NH_4^+-N	20.8	21.6
TKN	32.9	34.6

Conclusions & Recommendations

5.1 Conclusions

Different anoxic and anaerobic hydraulic retention times were evaluated to up-concentrate the domestic wastewater via Biosorption-Sedimentation Process. The continuous lab scale sewage up-concentration experiments proved the clear advantage of T_{ano}/T_{an} of 60 minutes/60 minutes, considering the both up-concentration (in terms of COD and TP) and minimum ammonium-nitrogen ($NH4^+$-N) concentration in the concentrated effluent stream. The theoretical maximum potential of energy within the effluent was calculated as 15 kWh/m^3 of domestic wastewater. With the addition of up-concentration step in future treatment plants can be net energy positive treatment plant.

5.2 Recommendations

- ☐ To run the system with same operational parameters with real domestic wastewater and to compare the results.
- ☐ To check how much biogas will be produced with the concentrated stream by adding anaerobic digestion (AD) reactor.
- ☐ To couple this system with anaerobic membrane reactor to further optimize the system.

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