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Microsieving as a Primary Treatment for Biological Nitrogen Removal from Municipal Wastewater

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Abstract

There has been an increased interest in alternative carbon diversion technologies in wastewater treatment to improve the efficiency and performance of primary treatment, increase treatment capacity, and minimize overall energy consumption, especially in geographies with limited space for expansion. Microsieving technologies like the rotating belt filters (RBFs) have emerged as a promising primary solids separation alternative to primary clarification. This research was conducted to study the implications of retrofitting existing wastewater treatment plants (without primary treatment) with RBF technology.

In order to fully evaluate the impact of RBF in water resource recovery facilities, it is paramount to investigate the unique characteristics of the more fibrous material removed by microsieving, cellulose, mostly in the form of toilet paper, which is a major component of the particulates in raw municipal wastewater. To date, a validated method for cellulose quantification in wastewater and sludge matrices was unavailable. This research demonstrated that the Schweitzer-reagent method is a very robust and reliable cellulose quantification method in light of its reproducibility and accuracy. Sludge from the RBF was observed to contain 37±1 % cellulose (on dry basis), whereas primary clarifier sludge contained 18±0.2 % cellulose (on dry basis) which confirmed that the RBF captures the cellulose more efficiently than the primary clarifier. The contribution from this work would have great implications on wastewater research in understanding the fate of toilet-paper-cellulose, and its impact on biosolids management given the already emerging trend to increase sustainability and resource recovery.

When looked in the context of the impact of the RBF on activated sludge processes, RBF effluent was compared with raw wastewater and primary clarifier effluent. This was accomplished using respirometric techniques to identify the most influential biokinetic parameters required for model simulations. The raw wastewater was predominantly biodegradable where 71% of the TCOD was observed to be biodegradable. Primary clarifier and RBF treatment increased the biodegradable fraction to 80% and 74%, respectively, by removing inert particulates by settling and microsieving, respectively. As

expected, microsieving and settling do not impact the soluble components in the wastewaters. The fractionation of the particulate components was dictated by the primary treatment suspended solids removal efficiency and was observed to be comparable for the RBF effluent and the primary clarifier effluent. The implementation of different COD fractions and kinetic coefficients of the RBF effluent would improve the model simulations for design, control, and optimization of biological wastewater treatment processes employing RBF as a primary treatment.

In addition, the results from this study established that the RBF offers an alternative level of treatment (to primary clarification), which removes particulate solids, without impacting nitrification and denitrification processes with total nitrogen removal efficiency ranging from 68%-73% for medium-strength wastewater. Upon modeling (using GPS-X) to predict performance for high-strength wastewater, it was observed that within the TSS removal of 27%-70% by the RBF, biological nitrogen removal was not adversely affected (79% total nitrogen removal). Moreover, the overall primary and biological sludge production by a wastewater resource recovery facility employing an RBF as primary treatment was found to be 9% lower than the one with primary clarification. Chemically-enhanced-RBF treatment was observed to be ideal for plants trying to achieve BOD and ammonia limits; however, excessive removal of carbon compromised nitrogen removal efficiency (30% total nitrogen removal), especially with low-strength wastewaters.

The findings of this work would instigate further research on RBF technology for successful integration as a primary treatment alternative in wastewater resource recovery facilities.

Keywords

Carbon diversion; Cellulose; COD fractionation; Microsieving; Nitrogen removal; Primary treatment; Respirometry; Rotating belt filter; Schweitzer reagent; Toilet paper; Sludge; Wastewater

Co-Authorship Statement

This PhD thesis contains material that is published, 'under review' or in preparation for submission in peer reviewed journals as listed below.

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The primary author of this chapter was Gupta under the supervision of Dr. Nakhla and Dr. Santoro. The experimental plan was developed by Gupta with guidance from Dr. Santoro and Dr. Nakhla while the execution of experiments, data collection and analysis, and drafting the manuscript was conducted by Gupta. Feedback on the manuscript was received from other co-authors.

Chapter 4: Evaluation of COD Fractionation and Biokinetic Parameters of Microsieved Wastewater

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The experimental plan was developed by Gupta with guidance from Dr. Nakhla and Dr. Santoro while the execution of experiments, data collection and analysis, and drafting the manuscript was conducted by Gupta. Ganesh Ram Dutt Sridhar assisted with data collection.

Chapter 5: Microsieving Raw Wastewater for Nitrogen Removal and Control in Wastewater Resource Recovery Facilities

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The primary author of this chapter was Gupta under the supervision of Dr. Nakhla and Dr. Santoro. The experimental plan was developed by Gupta with guidance from Dr. Santoro and Dr. Nakhla while the execution of experiments, data collection and analysis, and drafting the manuscript was conducted by Gupta. Feedback on the manuscript was received from other co-authors. The GPSx modeling simulations were conducted by Dr. Dang Ho.

Chapter 6: Evaluation of Chemically-Enhanced Microsieving on Nitrogen Removal in Wastewater Resource Recovery Facilities

In preparation for submission to *Water Research*.

The experimental plan was developed by Gupta with guidance from Dr. Nakhla and Dr. Santoro while the execution of experiments, data collection and analysis, and drafting the manuscript was conducted by Gupta. Ganesh Ram Dutt Sridhar assisted with data collection. Trojan Research team assisted with sample preparation.

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Medhavi Gupta

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“Nothing in the world can take the place of persistence. Talent will not; nothing is more common than unsuccessful men with talent. Genius will not; unrewarded genius is almost a proverb. Education will not; the world is full of educated derelicts. Persistence and determination alone are omnipotent. The slogan "press on" has solved and will always solve the problems of the human race.”

- *Calvin Coolidge*

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List of Abbreviations and Symbols

ASM	Activated Sludge Model
ATP	adenosine triphosphate
Anammox	anaerobic ammonium oxidation
AOB	ammonium-oxidizing bacteria
ATU	allythiourea
BNR	biological nutrient removal
BOD	biochemical oxygen demand (mg/L)
BOD_5	5-day biochemical oxygen demand (mg/L)
cBOD	carbonaceous biochemical oxygen demand (mg/L)
CEPT	chemically-enhanced primary treatment
CE-RBF	chemically-enhanced rotating belt filter
COD	chemical oxygen demand (mg/L)
CON	colloidal organic nitrogen (mg/L)
CSO	combined sewer overflow
DP	degree of polymerization
DO	dissolved oxygen (mg/L)
EBPR	enhanced biological phosphorous removal
FA	free ammonia
FNA	free nitrous acid
HMF	5-hydroxymethylfurfural
HS-RWW	high-strength raw wastewater
ISS	inert suspended solids (mg/L)

MBR	membrane bioreactor
MBBR	moving bed biofilm reactor
MLE	Modified Ludzack Ettinger
nbCOD	nonbiodegradable COD (mg/L)
nbsCOD	nonbiodegradable soluble COD (mg/L)
nbpCOD	nonbiodegradable particulate COD (mg/L)
nbVSS	nonbiodegradable volatile suspended solids
NOB	nitrite-oxidizing bacteria
NREL	National Renewable Energy Laboratory
NUR	nitrate uptake rate
OHO	ordinary heterotrophic organisms
ON	organic nitrogen
OP	organic phosphorous
OU	oxygen uptake
OUR	oxygen uptake rate (mg/L)
pbCOD	particulate biodegradable chemical oxygen demand (mg/L)
PC	primary clarification
PCE	primary clarifier effluent
PON	particulate organic nitrogen
RAS	return activated sludge
RWW	raw wastewater
rbCOD	readily biodegradable COD (mg/L)
rbsCOD	readily biodegradable soluble COD (mg/L)
RBF	rotating belt filter
RBFE	rotating belt filter effluent

RWW	raw wastewater
sbCOD	slowly biodegradable COD (mg/L)
SBR	sequential batch reactor
SCOD	soluble chemical oxygen demand (mg/L)
SNR	specific nitrification rate (mg/g.d)
SDNR	specific denitrification rate (mg/g.d)
SMP	soluble microbial product (mg/L)
SON	soluble organic nitrogen (mg/L)
S_o/X_o	substrate-to-biomass ratio
SRT	solids retention time (d)
TCOD	total chemical oxygen demand (mg/L)
TOC	total organic carbon (mg/L)
TKN	total Kjeldahl nitrogen (mg/L)
TN	total nitrogen (mg/L)
TP	total phosphorus (mg/L)
TS	total solids (mg/L)
TDS	total dissolved solids (mg/L)
TSS	total suspended solids (mg/L)
UASB	upflow anaerobic sludge blanket
VFA	volatile fatty acids (mg/L)
VSS	volatile suspended solids (mg/L)
WEF	Water Environment Federation
WRRF	wastewater resource recovery facility
WWTP	wastewater treatment plant
$XBOD_5$	particulate BOD_5 (mg/L)

XCOD	particulate chemical oxygen demand (mg/L)
b_H	decay rate (d^{-1})
C_T	total chemical oxygen demand (mg/L)
C_S	biodegradable chemical oxygen demand (mg/L)
C_I	non-biodegradable chemical oxygen demand (mg/L)
μ_n	specific growth rate of nitrifying bacteria (d^{-1})
μ_{nm}	maximum specific growth rate of nitrifying bacteria (d^{-1})
μ_{max}	maximum specific growth rate (d^{-1})
K_n	substrate concentration at one-half the maximum specific substrate utilization rate (mg/L)
K_o	half-saturation concentration for dissolved oxygen (mg/L)
K_{dn}	decay coefficient for nitrifying bacteria (d^{-1})
k_H	hydrolysis coefficient (d^{-1})
K_S	substrate half-saturation coefficient (mg/L)
S_S	readily biodegradable chemical oxygen demand (mg/L)
S_H	rapidly hydrolysable chemical oxygen demand (mg/L)
S_I	soluble inert chemical oxygen demand (mg/L)
X_S	slowly biodegradable chemical oxygen demand (mg/L)
X_H	heterotrophic biomass (mg/L)
X_I	particulate inert chemical oxygen demand (mg/L)
Y_H	heterotrophic biomass yield (mg COD/mg COD)
Y_{obs}	observed biomass yield (mg COD/mg COD)

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Chapter 1

1 Introduction

1.1 Rationale

While nutrients are essential constituents of living organisms, nutrient removal is essential to maintain their natural cycle within the ecosystem, following humankind influence [Ambulkar, 2017]. The goals of this research were motivated by the increased concerns regarding nutrient discharges from municipal wastewater treatment plants. Increased eutrophication and ecological concerns in receiving surface waters have caused regulators to reduce nutrient discharge limits, to as low as <1.5 to 3 mg total nitrogen/L and <0.07 mg total phosphorous/L [Oleszkiewicz and Barnard, 2006]. The need to meet compliance with these stringent regulations has stirred research to optimize current nutrient removal processes without additional expenditure or higher operational cost. [Rossle and Pretorius, 2001; EPA, 2008; Oleszkiewicz and Barnard, 2006].

Carbon availability is essential to promote conventional denitrification and enhanced biological phosphorous removal (EBPR) in biological nutrient removal (BNR) plants. Moreover, the quality of the carbon provided is equally as important which has led to two different design strategies. The first option relies on the use of a primary clarification step, which is specified to divert slowly biodegradable carbon in the form of settleable particles while allowing readily (soluble) biodegradable carbon to be exploited in the downstream biological treatment process, specifically in the denitrification stage [Tchobanoglous et al., 2003]. Additionally, primary clarification reduces the solids loading, aeration energy requirements, and biological sludge production in nutrient removal processes. However, excessive carbon removal by primary clarifiers causes incomplete nitrogen and phosphorous removal. In such cases, external carbon dosing is incorporated, inevitably increasing operational costs. Alternatively, in order to maximize internal wastewater carbon utilization, primary clarifiers are omitted which undesirably causes solids overloading in the secondary clarifiers together with higher sludge production and increased aeration costs [Ubay-Cokgor et al., 2005; Gori et al., 2013; Spellman, 2013].

Driven by the shift in municipal treatment goals from merely environmental protection towards resource recovery, carbon diversion processes are gaining popularity. In particular, high rate primary solids removal processes capable of achieving particulate removal, in a compact footprint, are receiving considerable attention as they combine the advantages of both design philosophies discussed above [Franchi and Santoro, 2015; Caliskaner et al., 2014; Oleszkiewicz, 2015]. The rotating belt filter (RBF) is an emerging technology for primary treatment where removal of particulates from wastewater are achieved by microsieving. The RBF uses a filtermesh mounted on an inclined rotating belt to microsieve solids from wastewater. RBFs may represent an ideal primary separation option since in addition to minimal space requirement, they address two design requirements for BNRs, i.e. no removal of readily biodegradable carbon and maximum diversion of slowly biodegradable particulates, which is typically enriched in cellulose, a carbon fraction known to have limited biodegradability under both anaerobic and aerobic conditions [Ruiken et al., 2013; Paulsrud et al., 2014; Sarathy et al., 2015; Ghasimi, 2016]. Therefore, in order to enable a successful integration of RBF into BNR schemes, it is critical to evaluate the carbon fractionation of RBF effluents as well as its impact on downstream biological treatment process. Additionally, quantitative assessment of cellulose content in RBF sludge would aid in better understanding of the diversion of slowly biodegradable particulates.

1.2 Objectives

This research was conducted to study the implications of retrofitting existing wastewater treatment plants (without primary treatment) with RBF technology and comparing it with conventional primary clarification. Figure 1-1 illustrates the scheme of RBF primary treatment to enhance BNR. While the overall objective of this research was to evaluate the quality of effluent from the RBF technology for biological nutrient removal, the sludge obtained from the RBF was characterized for cellulose content to better understand the carbon fractionation. The specific research objectives of this work are outlined as follows:

I. To assess and validate different methods to quantify cellulose in wastewater and sludge, as well as quantitatively compare the cellulose content in RBF sludge and primary clarification sludge

II. To investigate the carbon fractionation and biokinetic parameters of RBF effluent, and compare it with primary clarifier effluent using aerobic respirometry and batch denitrification tests

III. To study the impact of RBF on biological nitrogen removal from municipal wastewater in sequential batch reactors in terms of nitrification-denitrification

IV. To study the impact of chemically-enhanced RBF on biological nitrogen removal from municipal wastewater in sequential batch reactors in terms of nitrification-denitrification

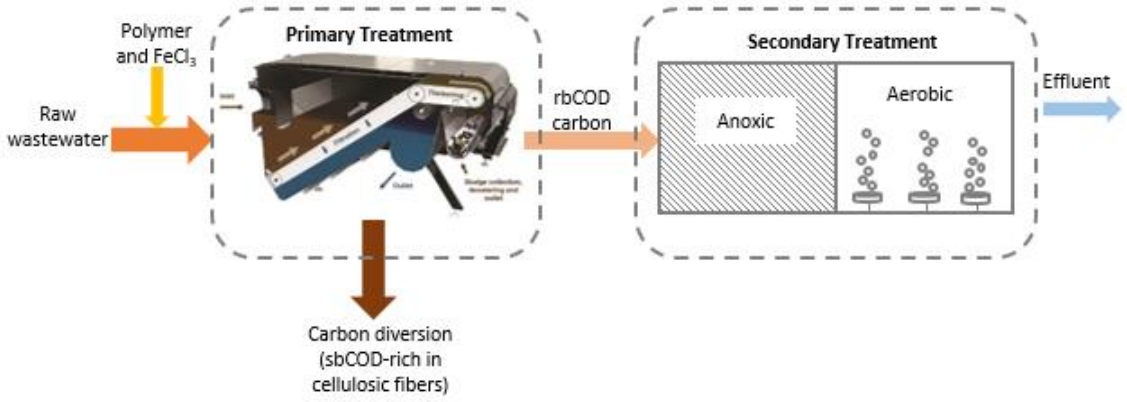

Figure 1-1. Rotating belt filter (RBF) scheme to enhance Biological Nitrogen Removal

1.3 Thesis Organization

Chapter 1 presents an overview of the thesis and the rationale behind assessing newly emerging microsieving technology as an alternative to primary treatment at wastewater resource recovery facilities. It summarizes the most relevant literature to this research as

well as the specific research objectives. Chapter 2 provides a comprehensive literature review on various primary treatment processes/technologies including primary clarification and RBF, as well as theory of respirometry and biological nitrogen removal in wastewater, and review of relevant research studies.

Chapter 3 is a research article entitled "*Experimental Assessment and Validation of Quantification Methods for Cellulose Content in Municipal Wastewater and Sludge*". The objective of this work was to compare the different cellulose measurement methods and to validate the most reliable method to accurately quantify cellulose in a complex matrix of wastewater and sludge. Four different methods were tested including dilute-acid hydrolysis, concentrated acid hydrolysis, enzymatic hydrolysis, and the Schweitzer method. The main drive for this work was to quantitatively determine the cellulose content in RBF and primary clarification sludges.

Chapter 4 is a research article entitled "*Evaluation of COD Fractionation and Biokinetic Parameters of Microsieved Wastewater*", that discusses the fractionation of different COD fractions in raw wastewater, primary clarifier effluent, and RBF effluent to better understand the implications of using RBF for primary treatment as an alternative to primary clarification.

Chapter 5 is a research article entitled "*Microsieving Raw Wastewater for Nitrogen Removal and Control in Wastewater Resource Recovery Facilities*". In this study, the impact of primary treatment by RBF on biological nitrogen removal was evaluated and compared against primary clarification. Chapter 6 is a research article entitled "*Evaluation of Chemically-Enhanced Microsieving for Nitrogen Removal in Wastewater Resource Recovery Facilities*". In this study, the impact of chemically-enhanced RBF on biological nitrogen removal was evaluated.

Finally, Chapter 7 summarizes the major findings of this research study together with future recommendations.

1.4 Thesis Format

This thesis is prepared in the integrated-article format according to the specifications provided by the School of Graduate and Postdoctoral Studies at the University of Western Ontario. Chapter 3 of this thesis is "under review" in *Environmental Science and pollution Research*. Chapter 4 has been prepared to be submitted to *Water Environment Research* journal. Chapter 5 is "under review" in *Environmental Technology* journal. Chapter 6 has been prepared to be submitted to *Water Research* journal.

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Chapter 2

2 Literature Review

2.1 Organics and Nutrients in Wastewater

High nutrient concentrations in the effluent discharge to surface waters can cause severe ecological problems including eutrophication, ammonia toxicity, etc. Moreover, environmental and public health concerns rise when partially-treated or untreated wastewater is released to receiving water bodies that are eventually used as recreational bodies or water supplies. In fact, accumulation of organics could lead to septic conditions, which could promote the production and release of greenhouse gases as well as proliferation of pathogenic and non-pathogenic microorganisms [WEF, 2005a]. For the above-mentioned reasons, wastewater treatment is crucial for effective control and management of environmental and health impacts and the release of contaminants in the environment.

2.1.1 Organics

Organic compounds are one of the major concerns in wastewater treatment and typically consist of proteins, carbohydrates, and oils and fats, as well as urea and different synthetic organic molecules. Organic matter in wastewater (as well as inorganic matter) is typically measured as chemical oxygen demand (COD) which is the amount of oxygen consumed for decomposition of organic matter (and oxidation of inorganic matter). Biodegradable organic matter is measured as biochemical oxygen demand (BOD) [Tchobanoglous et al., 2003] that is a measure of the amount of oxygen consumed by microbial oxidation.

Figure 2-1 illustrates the fractionation of COD in wastewater. Some fractions of the COD are nonbiodegradable (nbCOD) and pass through secondary treatment unaffected; the nonbiodegradable soluble COD (nbsCOD) leaves with the secondary effluent and the nonbiodegradable particulate COD (nbpCOD) ends up in the sludge. Readily biodegradable COD (rbCOD) is usually soluble (rbsCOD) and is assimilated by the biomass. Particulate biodegradable COD must first be solubilized and thus translates to

slower removal rates. rbCOD consists of complex COD that can be fermented to volatile fatty acids (VFAs). The BOD/COD ratio for municipal wastewater is typically in the range from 0.3 to 0.8 [Tchobanoglous et al., 2003; Henze et al., 2008]. Table 2-1 shows the typical component ratios in municipal wastewater [Henze et al., 2008].

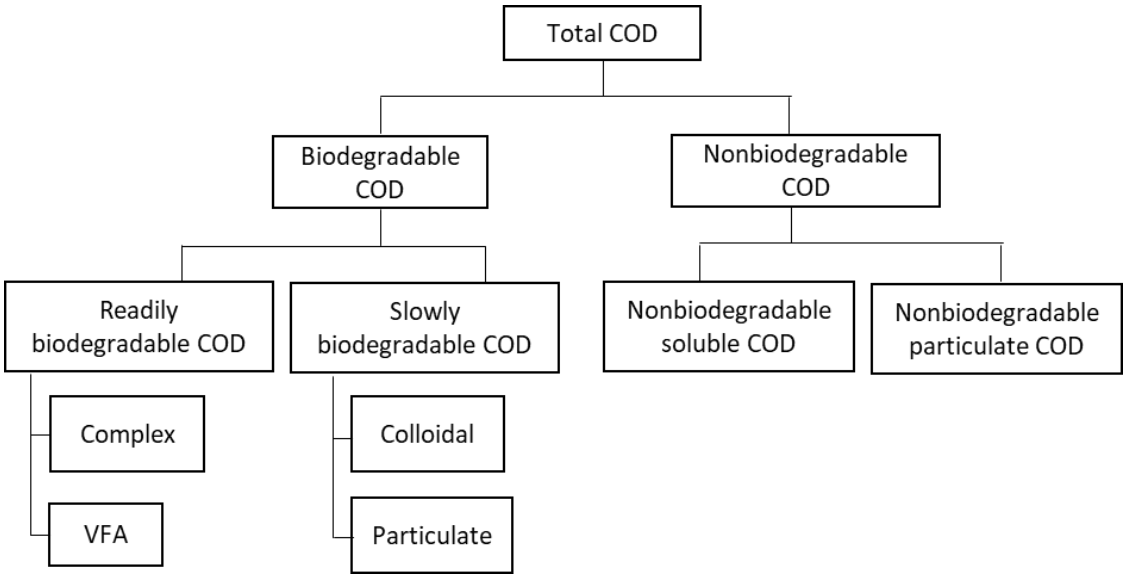

Figure 2-1. Fractionation of COD in wastewater

Table 2-1. Typical ratios of municipal wastewater [Henze et al., 2008]

Ratio	High	Medium	Low
COD/BOD	2.5-3.5	2.0-2.5	1.5-2.0
VFA/COD	0.08-0.12	0.04-0.08	0.02-0.04
COD/TOC	3.0-3.5	2.5-3.0	2.0-2.5
COD/VSS	1.6-2.0	1.4-1.6	1.2-1.4

The recent paradigm shift from wastewater treatment plants (WWTPs) to wastewater resource recovery facilities (WRRFs) has led to the evolution of treatment process and technologies. The introduction of advanced treatment technologies has stimulated the need for a deeper understanding of the different COD components in municipal wastewater, and their behavior in the treatment processes.

2.1.2 Solids

Total solids (TS) content in wastewater is the most important physical characteristic and can be divided into total suspended (TSS) and dissolved (TDS) solids (Fig. 2-2). Suspended solids are usually a portion of the TS retained on a filter paper of specific pore size (usually 1.2 μm) after being dried at 105°C. 60% of suspended solids in municipal wastewater are settleable. The solids contained in the filtrate that passed through the filter paper consists of dissolved and colloidal solids. The solids contained in wastewater are either fixed (inert) or volatile (biodegradable). The volatile fraction contributes to the BOD, nitrogen, and phosphorous, and the VSS/TSS is typically 0.6-0.8 [WEF, 2005a; Tchobanoglous et al., 2003, Henze et al., 2008].

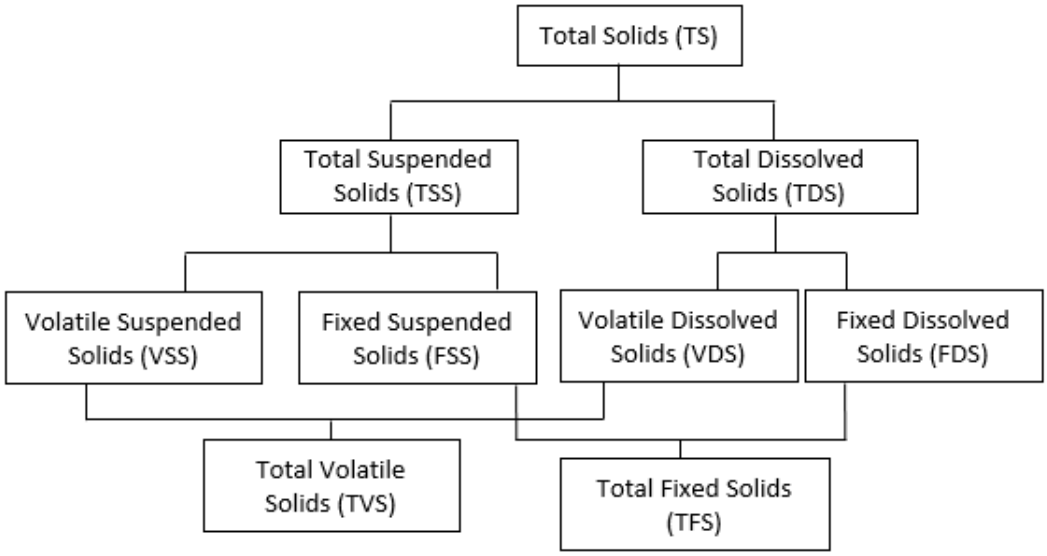

Figure 2-2. Solids fractionation [Tchobanoglous et al., 2003]

2.1.2.1 Toilet paper in wastewater solids

Of the insoluble solids in wastewater treatment plant influents, cellulose, in the form of toilet paper, has been reported to be a major component which inadvertently ends up in sewage sludge [Edberg and Hofsten 1975; Verachtert et al. 1982]. Toilet paper is a widely used hygiene product in developed countries. Toilet paper consumption in western countries has been reported to be around 10-14 kg per capita per year, which makes up

around 30%-50% of the suspended solids in influent wastewater [Ramasamy et al., 1981; Ghasimi, 2016; Chen et al., 2017].

Cellulose, $(C_6H_{10}O_5)_n$, is considered a complex carbohydrate very similar to starch, and is a linear polymer of β-1,4-glycosidic bond linked with D-glucose units [Chen et al., 2017]. Cellulose is a valuable resource which if recovered can be used for various other applications such as production of fuels and chemicals, building materials, bioplastics, flocculants etc. [Rinaldi and Schüth, 2009]. Microsieving technologies (e.g., Rotating Belt Filter), have shown significant potential for cellulose recovery from raw wastewater [Ruiken et al., 2013] with potential downstream increase in biological processing capacity. Moreover, due to the low rate of cellulose biodegradation under aerobic conditions, the removal of cellulose and other fiber-like material is expected to lead to additional operational savings such as lower aeration energy consumption and secondary sludge production. Due to the above-mentioned reasons, it is essential to extend the characterization of the solids to cellulose content to better understand its fate in wastewater treatment facilities.

2.1.3 Nitrogen

Nitrogen and phosphorous are the inorganic chemical constituents of concern in wastewater and are essential to the growth of microorganisms, commonly referred to as nutrients or biostimulants. The most important forms of nitrogen in wastewater are ammonia (NH_3), ammonium (NH_4^+), nitrogen gas (N_2), nitrite ion (NO_2^-), nitrate ion (NO_3^-), and organic nitrogen [Tchobanoglous et al., 2003; WEF, 2005a]. Figure 2-3 shows the fractionation of nitrogen in wastewater [Tchobanoglous et al., 2003]. Table 2-2 illustrates the typical nitrogen composition in wastewater [WEF, 2005a; Henze et al., 2008]. Wastewater treatment plants receive nitrogen in the form of total Kjeldahl nitrogen (TKN) of which 60% will be as ammonia (NH_4-N) and 40% in the organic form [WEF, 2005a]. Particulate biodegradable organic nitrogen (ON) consists of amino acids and proteins that are hydrolyzed to ammonium by bacterial decomposition in a process called ammonification [WEF, 2005a]. Soluble biodegradable nitrogen is easily assimilated by the microorganisms as a nitrogen source. The nonbiodegradable ON is present in soluble (SON), colloidal (CON), and particulate forms (PON), where SON and

CON exits in the secondary effluent whereas PON exits in waste sludge, respectively. The SON-CON is comprised of both influent-derived recalcitrant organic nitrogen, and plant-derived fraction produced during biomass decay (fraction of soluble microbial products (SMPs)) [Pagilla et al., 2011]. CON has been reported to range from 43% to 78% of the effluent total nitrogen (TN) whereas SON could range from 56% to 95% of the TN in the final effluent [Sattayatewa et al., 2010; Czerwionka et al., 2012]. Interestingly, it has also been reported that irrespective of influent and effluent TN concentration, the magnitude of effluent SON ranges from 0.5 to 2 mg N/L [Sattayatewa et al., 2010].

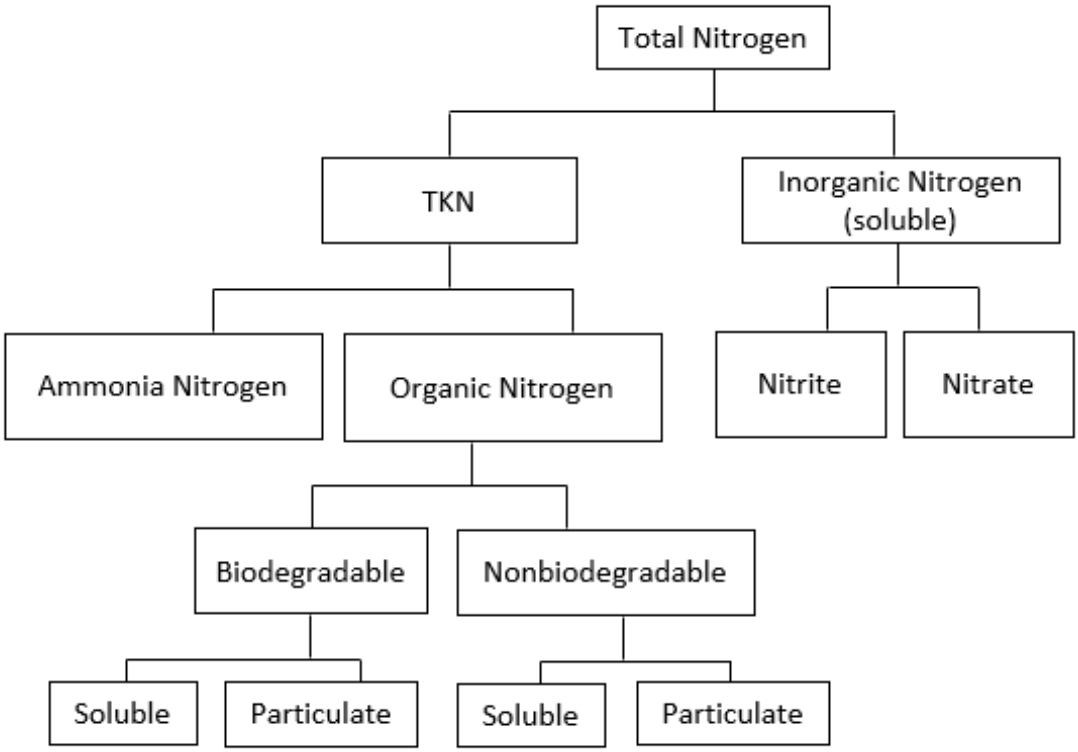

Figure 2-3. Nitrogen fractionation in wastewater [Tchobanoglous et al., 2003]

Table 2-2. Typical nitrogen forms and composition in wastewater [WEF, 2005a; Henze et al., 2008]

Parameter	High	Medium	Low
Total nitrogen (mg/L)	70	40	20
Organic (mg/L)	25	15	8
Free ammonia (mg/L)	45	25	12
Nitrites (mg/L)	0	0	0
Nitrates (mg/L)	0	0	0
COD/TN ratio	12-16	8-12	6-8
BOD/TN ratio	6-8	4-6	3-4

2.1.4 Phosphorous

Phosphorous is also an essential nutrient for the growth of algae and other microorganisms. The most important forms of phosphorous in aqueous solutions are orthophosphate (PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^-$, H_3PO_4), polyphosphate (condensed phosphates), and organic phosphate (phospholipids and nucleotides) [WEF, 2005a; Tchobanoglous et al., 2003]. Phosphorous in wastewater can be classified as inorganic and organic phosphorous (Fig. 2-4). Orthophosphate (also known as reactive phosphorous) and polyphosphates (also known as acid hydrolysable phosphorous) are the inorganic forms of phosphorous. Polyphosphates are transformed into orthophosphate upon acid addition. Orthophosphate accounts for 70 to 90% of total phosphorous (TP) which is readily assimilated by microorganisms without further breakdown. Soluble and particulate organic phosphorous (OP) can be further classified into biodegradable and non-biodegradable. The particulate organic phosphorous (biodegradable and nonbiodegradable fractions) is typically precipitated and removed in the sludge. The soluble biodegradable organic phosphorous can be hydrolyzed to orthophosphates. Chemical phosphorous removal is effective in removing soluble reactive and acid-hydrolysable phosphorous, and particulate OP, but not particulate acid-hydrolysable phosphorous and soluble OP. The soluble nonbiodegradable OP can range from 2% to 11% of the total final effluent phosphorous [Gu et al., 2011]. Table 2-3 shows the typical

phosphorus concentrations found in municipal wastewater [WEF, 2005a; Henze et al., 2008].

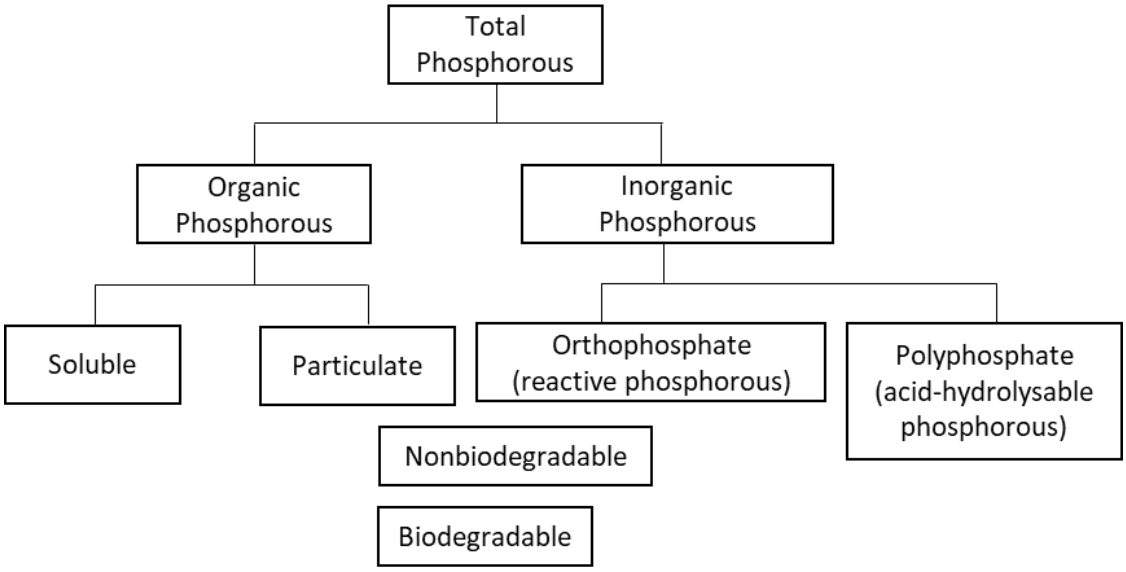

Figure 2-4. Phosphorous fractionation in wastewater [WEF, 2005a]

Table 2-3. Typical phosphorous composition in wastewater [WEF, 2005a; Henze et al., 2008]

Contaminant	High	Medium	Low
Total phosphorous (mg/L)	12	7	4
Organic (mg/L)	4	2	1
Inorganic (mg/L)	10	5	3
COD/TP ratio	45-60	35-45	20-35
BOD/TP ratio	20-30	15-20	10-15

2.2 Respirometry for Bioprocess Modelling

The biochemical definition of microbial respiration is the metabolic process in which electrons removed from the electron donor are transferred along the electron transport chain (Fig. 2-5), and eventually taken up by the ultimate electron acceptor. Energy, in the

form of adenosine triphosphate (ATP), is generated which is used for biomass growth, maintenance, and reproduction [Spanjers and Vanrolleghem, 2017]. Spanjers et al. [1998] defines respirometry as the "measurement and interpretation of the rate of biological consumption of an inorganic electron acceptor under well-defined experimental conditions" (Fig. 2.6) [Spanjers and Vanrolleghem, 2017]. In activated sludge processes (where oxygen is the ultimate electron acceptor), it is the biological oxygen consumption (also called respiration rate) that is directly associated with heterotrophic biomass growth and carbonaceous substrate removal [Vanrolleghem, 2002; Vitanza et al., 2016]. Fig. 2-7 illustrates the overall aerobic respiration by heterotrophic biomass [Spanjers and Vanrolleghem, 2017].

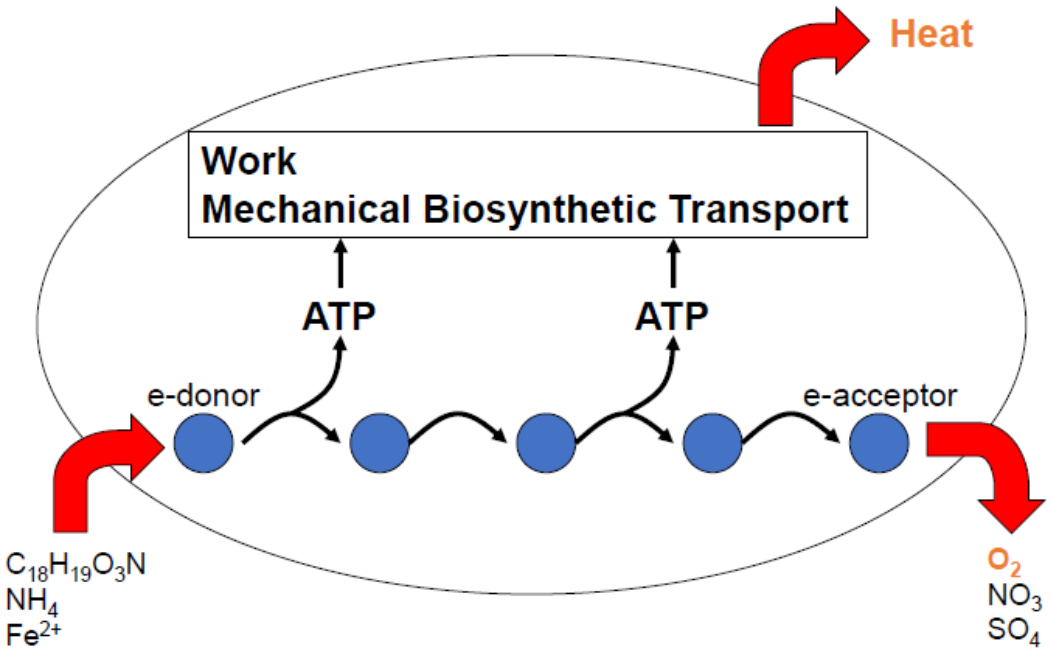

Figure 2-5. Microbial respiration: Electron Transport Chain [Spanjers and Vanrolleghem, 2017]

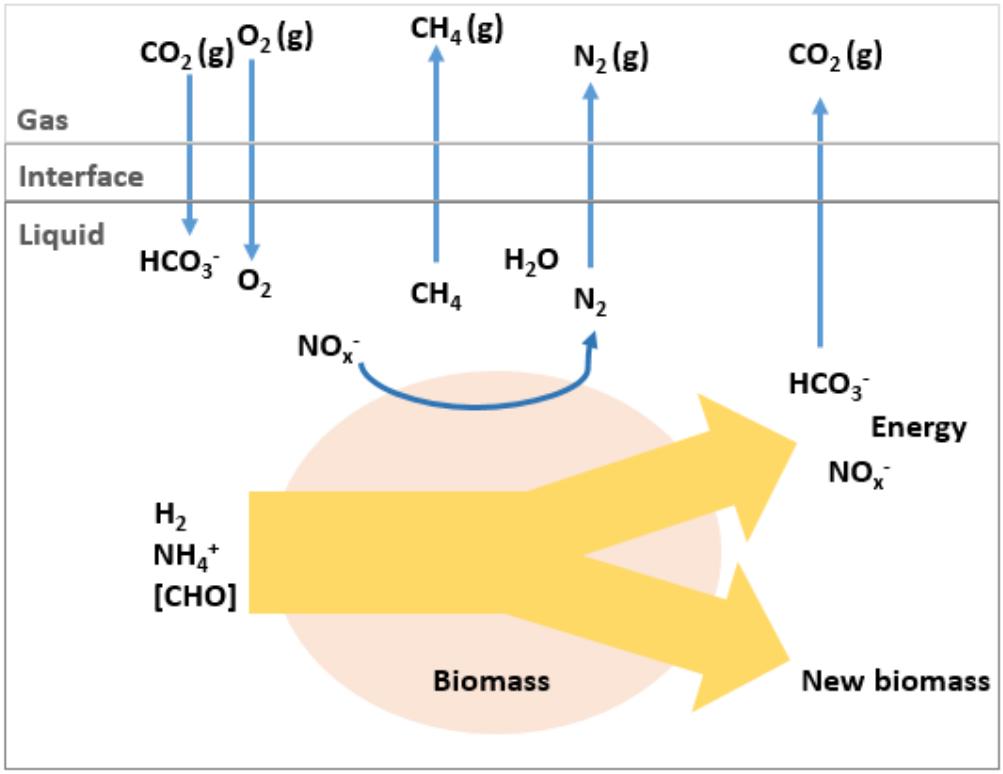

Figure 2-6. Basics of respiration [adapted from Spanjers and Vanrolleghem, 2017]

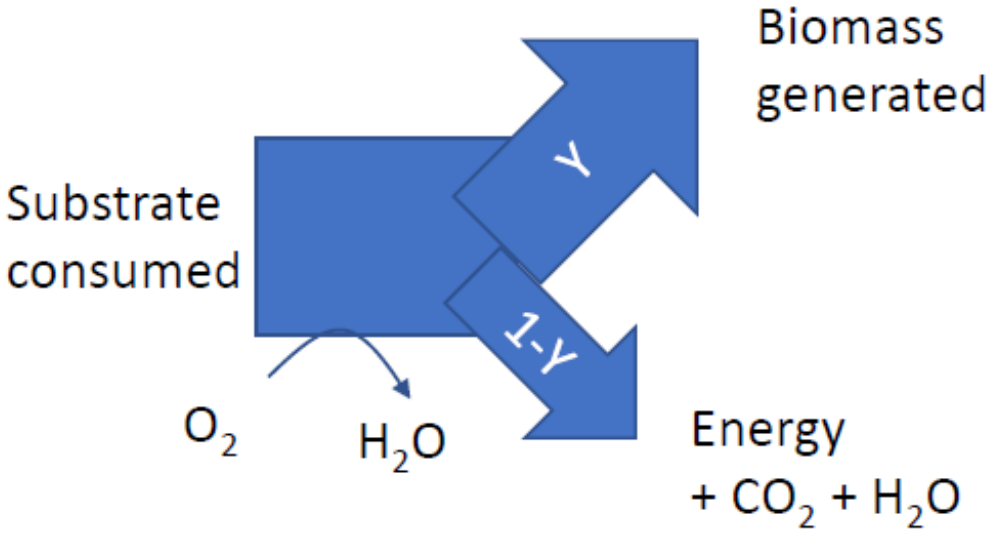

Figure 2-7. Schematic illustration of aerobic respiration by heterotrophic biomass [Vanrolleghem, 2002]

Respirometry is one of the oldest (as early as 1920s) techniques used to determine COD fractionation, kinetic parameters, and stoichiometric coefficients, which are essential inputs to all multicomponent models such as the Activated Sludge Model (ASM) [Rahman and Islam, 2015]. These models are widely used for design, operation, control, troubleshooting, upgrading, modelling, and optimization of biological wastewater treatment processes [Spanjers and Vanrolleghem, 1995; Gernaey et al., 2001; Xu et al., 2006; Liwarska-Bizukojc and Biernacki, 2010; Liwarska-Bizukojc and Ledakowicz, 2011; Torretta et al., 2014]. Respirometers are instruments that measure respiration rate or oxygen uptake rate (OUR).

Modelling goals and process dictate the level of characterization required. Wastewater total COD (C_T) can be fractionated to various biodegradable (C_S) and inert (C_I) (non-biodegradable) fractions, where these fractions can be soluble (S) or particulate (X) in nature as illustrated in Fig. 2-1. The biodegradable fraction incudes readily biodegradable COD (S_S), rapidly hydrolysable COD (S_H), and slowly biodegradable COD (X_S) [Orhon et al., 1994; Tran et al., 2015].

In a respirometry test, OUR profiles are generated from an aerated batch reactor fed with a pre-determined substrate-to-biomass ratio (S_O/X_O) (typically 4 mg COD/mg VSS). To determine the various COD fractions and the kinetic parameters, a series of batch tests are performed on different fractions of wastewater and activated sludge: (i) unfiltered (raw) wastewater, (ii) filtered (0.45 µm) wastewater, (iii) mixed-liquor alone [Xu et al., 2006; Tran et al., 2015]. The equations required to determine the key COD fractions and kinetic parameters are outlined in Table 2.4 [Xu et al., 2006], of which Y_H, μ_{max}, b_H, and K_S, associated with ordinary heterotrophic organisms (OHOs), have been observed to be the most *influential* parameters for model calibration [Liwarska-Bizukojc and Biernacki, 2010]. Table 2-5 provides a summary of kinetic parameters, whereas Table 2-6 provides a summary of the typical COD fractionation in domestic wastewater.

The S_S and Y_H can be determined from the OUR profile of the filtered wastewater test based on Eq. 2.1 and 2.2. The Y_H is calculated by plotting net oxygen consumption and SCOD reduction (Fig. 2-8a; Eq. 2.1). The OUR during S_S reduction is approximately

constant, and once S_S is completely depleted, the OUR drops. The oxygen consumption before this drop in OUR is used to calculate S_S (Fig. 2-8b; Eq. 2.2). μ_{max} is also determined using filtered wastewater in accordance with Eq. 2.3 (Fig. 2-9a). b_H is determined using OUR profile of seed sludge-only test on the basis of Eq. 2.4 (Fig. 2-9b). X_H is calculated from the unfiltered wastewater-only test in accordance with Eq. 2.5, where f_e (inert COD from endogenous respiration) is assumed to be 0.2 g COD/g COD [Orhon et al., 1995; Xu et al., 2006]. S_I and X_I can be determined using the method described by Orhon et al. [1994] which involves running sequential batch reactors (SBRs) with filtered wastewater, unfiltered wastewater, and glucose, at a solids retention time of infinity to deplete all the biodegradable COD (Fig. 2-10). The residual COD in the glucose SBR is an estimate of soluble microbial products, and accordingly using Eq. 2.7, S_I can be calculated. Similarly, the unfiltered SBR test is used to determine X_I using Eq. 2.8. X_S and S_H fraction can be determined based on particulate COD balance (Eq. 2.6) and soluble COD balance (Eq. 2.9), respectively [Xu et al., 2006].

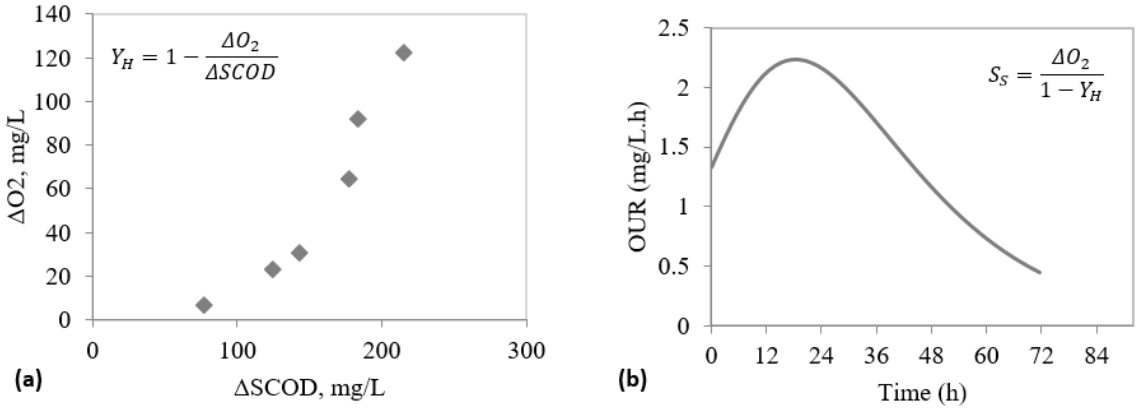

Figure 2-8. Determination of (a) Yield coefficient, (b) Readily biodegradable COD

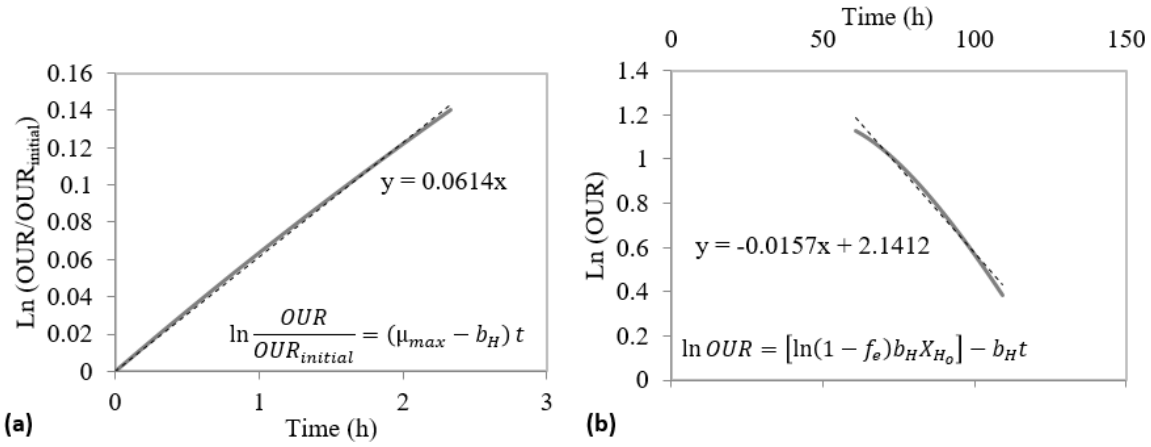

Figure 2-9. Determination of (a) Maximum heterotrophic growth rate, (b) Decay coefficient

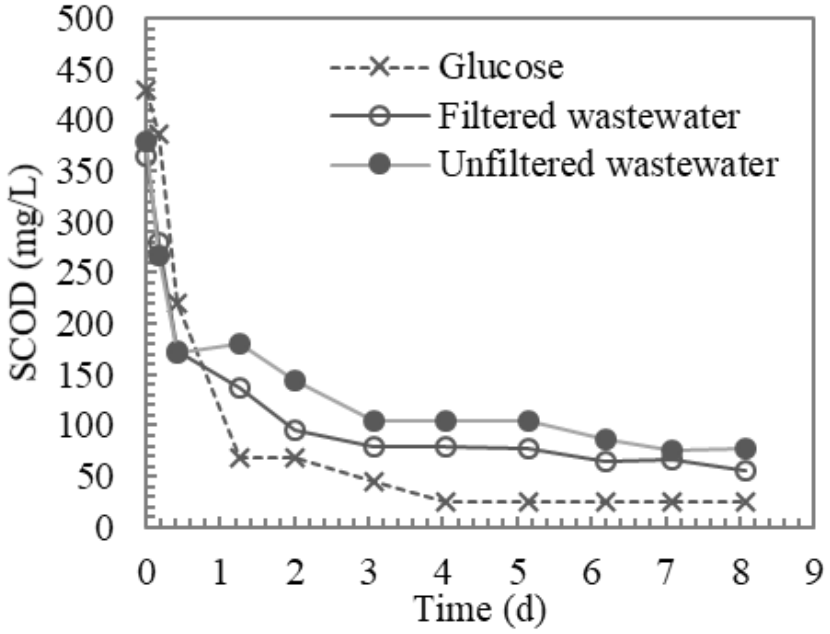

Figure 2-10. Determination of soluble and particulate inert COD

Maximum specific denitrification rate (SDNR) or nitrate uptake rate (NUR) tests are conducted to assess the anoxic heterotrophic activity for biomass characterization, where mixed liquor (4 g VSS/L) is mixed with NO_3-N (20 mg N/L) and acetate in excess (150 mg COD/L) while maintaining anoxic conditions [Spanjers and Vanrolleghem, 2017]. However, NUR tests can also be conducted to test the quality of carbon source in

wastewater. The reduction in COD associated with nitrate utilization is used to estimate the biodegradable COD content of wastewater [Ubay-Cokgor et al., 1998; Tas et al., 2009]. Section 2.4.2.2 of this chapter goes over the concepts of denitrification in biological wastewater treatment in detail as well as the typical ranges of SDNR reported in the literature.

Table 2-4. Equations to determine COD fractions and biokinetic parameters by using respirometric methods

Parameter	Equation	Eq.
Yield coefficient, Y_H	$Y_H = 1 - \frac{\Delta O_2}{\Delta SCOD}$	(2.1)
Readily biodegradable COD, S_S	$S_S = \frac{\Delta O_2}{1 - Y_H}$	(2.2)
Maximum heterotrophic growth rate, μ_{max}	$\ln \frac{OUR}{OUR_{initial}} = (\mu_{max} - b_H)\, t$	(2.3)
Endogenous decay coefficient, b_H	$\ln OUR = \left[\ln(1 - f_e) b_H X_{H_o}\right] - b_H t$	(2.4)
Heterotrophic biomass COD, X_{H_o}	$OUR_{initial} = \frac{1 - Y_H}{Y_H} \mu_{max} X_{H_o} + (1 - f_e)\, b_H X_{H_o}$	(2.5)
Substrate half-saturation coefficient, K_S	Parameter estimation	
Hydrolysis constant, k_H	Parameter estimation	
Slowly biodegradable COD, X_S	Parameter estimation or calculated by particulate COD balance: $X_S = C_T - S_T - X_I - X_H$	(2.6)
Soluble inert COD, S_I	$S_I = S_{R_1} - S_{P_G}$ Determined in SBRs with θ=∞ when S_S=0; where S_{R1} is residual soluble substrate in filtered wastewater reactor, and S_{PG} is the residual soluble inert microbial products in the glucose reactor.	(2.7)
Particulate inert COD, X_I	$X_I = C_T - C_S - S_I$ Determined in SBRs with θ=∞ when S_S=0, using unfiltered and filtered wastewater reactors.	(2.8)
Rapidly hydrolysable COD, S_H	$S_H = S_T - S_S - S_I$	(2.9)

Table 2-5. Summary of literature of kinetic parameters for activated sludge wastewater modeling

Parameter	Unit	Value	Reference
Y_H	mg cell COD/mg COD removed	0.58-0.61	Tran et al., 2015
		0.66	Ekame et al., 1986
		0.67	Ubay-Cokgor et al., 1998
		0.64-0.69	Orhon et al., 1994
		0.75-0.79	Strotmann et al., 1999
		0.63-0.67	Henze et al., 2000
μ_{max}	d^{-1}	3.6	Ekame et al., 1986
		4.5	Orhon et al., 1994
		3.5-6.5	Ubay-Cokgor et al., 2009
		3.5	Sperandio and Etienne, 2000
		1-6	Henze et al., 2000
K_S	mg COD/L	10-20	Orhon et al., 1994
		20	Henze et al., 2000
k_h	d^{-1}	2.2-3	Orhon et al., 1994
		2	Ubay-Cokgor et al., 1998
		3.1-3.8	Ubay-Cokgor et al., 2009
		3.5	Tas et al., 2009
		3.2	Sperandio and Etienne, 2000
		2-3	Henze et al., 2000
b_H	d^{-1}	0.2	Tas et al., 2009
		0.26	Sperandio and Etienne, 2000
		0.2-0.62	Henze et al., 2000

Table 2-6. Summary of literature of different COD fractions in domestic wastewater

Parameter	Unit	% of TCOD		Reference
		Domestic wastewater	Primary clarifier effluent	
S_S	mg COD/L	12%-22%		Ekame et al., 1986
		9% -10%	7%-33%	Ubay-Cokgor et al., 1998
		44.9%		Orhon et al., 1994
		10%	14%	Orhon et al., 1999; Ubay-Cokgor et al., 2009
		9%		Tas et al., 2009
			8.5%	Sperandio and Etienne, 2000
			10%-20%	Henze et al., 2000
		7%-29%		Yu et al., 2010
S_H	mg COD/L	27%	39%	Orhon et al., 1999
		13%		Tas et al., 2009
S_I	mg COD/L	3.8%		Orhon et al., 1994
		2%-5%	3%-20%	Ubay-Cokgor et al., 1998
		3%	4%	Orhon et al., 1999
		7%		Tas et al., 2009
			5%-12%	Henze et al., 2000
		2%-20%		Yu et al., 2010
X_I	mg COD/L	28.7%		Orhon et al., 1994
		13%-18%	4%-26%	Ubay-Cokgor et al., 1998
		7%	5%	Orhon et al., 1999
		16%		Tas et al., 2009
			10%-15%	Henze et al., 2000
		7%-20%		Yu et al., 2010
X_S	mg COD/L	22.6%		Orhon et al., 1994
		40%-62%	33%-60%	Ubay-Cokgor et al., 1998
		53%	38%	Orhon et al., 1999
		26%		Tas et al., 2009
			48%	Sperandio and Etienne, 2000
			30%-60%	Henze et al., 2000
		40%-62%		Yu et al., 2010
X_H	mg COD/L	20%	7%-25%	Ubay-Cokgor et al., 1998
			23%	Sperandio and Etienne, 2000
			5%-15%	Henze et al., 2000
		8%-20%		Yu et al., 2010

2.3 Solid Separation/Primary Processes

Primary sedimentation is the most widely used unit operation for removal of suspended solids from wastewater. However, there have been new innovative and emerging technologies to improve the efficiency and performance of primary treatment, and to meet the challenges of ever-changing nature of wastewater, population growth, changes in industrial processes, as well as aging infrastructure [EPA, 2013]. Additionally, carbon diversion technologies have been identified as one of the key wastewater treatment intensification approaches; among which enhanced primary treatment, filtration, and high-rate systems have been examined for sustainable increase in treatment capacity as well as energy optimization [WE&RF, 2016; Lema and Martinez, 2017].

2.3.1 Primary Clarification

Removal of settleable TSS in wastewater by gravitational settling is the conventional method used for primary treatment. Gravitational settling is also used in activated sludge setting tanks, combined sewer overflow (CSO), and for sludge thickening as well as storm water retention tanks [WEF, 2005b; Lema and Martinez, 2017]. While the solids underflow is an important consideration, the quality of primary effluent is of greater importance for operating expense of downstream processes [WEF, 2005b]. The performance of primary clarifiers is typically quantified based on TSS removal efficiency, calculated using Eq. 2.10:

$$TSS\ Removal\ Efficiency\ (\%) = 1 - \left(\frac{TSS_{primary\ influent}}{TSS_{primary\ effluent}}\right) \quad (2.10)$$

As TSS is removed in primary clarifiers, COD (or BOD) associated with the TSS gets removed, and similarly, BOD removal efficiency can be calculated using Eq. 2.10. Typically, primary clarifiers achieve 50% to 70% of TSS removal and 25% to 40% BOD removal [Tchobanoglous et al., 2003]. There are two types of primary clarifier, rectangular or circular tanks, both using mechanical cleaning. Figure 2-11 (a) and (b) shows a cross section of rectangular and circular clarifiers [Randall et al., 1992]. Flow is

horizontal in rectangular clarifiers as opposed to radial in circular clarifiers. Most primary clarifiers have a detention time of 1.5 to 2.5 hours depending on the wastewater flow and clarifier volume [Tchobanoglous et al., 2003]. Stacked clarifier, lamella clarifiers, and combined flocculator-clarifier are other designs of primary clarifiers. Stacked clarifiers have two or more tanks stacked on one another and therefore are smaller in footprint [Tchobanoglous et al., 2003]. Lamella clarifiers are conventional clarifiers with horizontal or inclined flat plates with varying cross-sections to increase surface area for settling [CH2M Hill, 2007]. Combined flocculator-clarifiers use inorganic chemicals or polymers to enhance settling also known as chemically-enhanced primary treatment (CEPT). Typically, iron or aluminum salts (e.g., ferric chloride or alum) are added in combination with polymer to improve performance by promoting settling of non-settleable TSS (colloidal TSS) [WEF, 2005b; Lema and Martinez, 2017]. TSS removal efficiencies can be increased from 55%-65% up to 75%-90%, and BOD removal efficiencies from 25%-40% to 50%-80% [CH2M Hill, 2007; Lema and Martinez, 2017]. Additionally, chemical addition to primary clarifiers enhances the removal of phosphorous, heavy metals, and hydrogen sulfide [WEF, 2005b; Lema and Martinez, 2017].

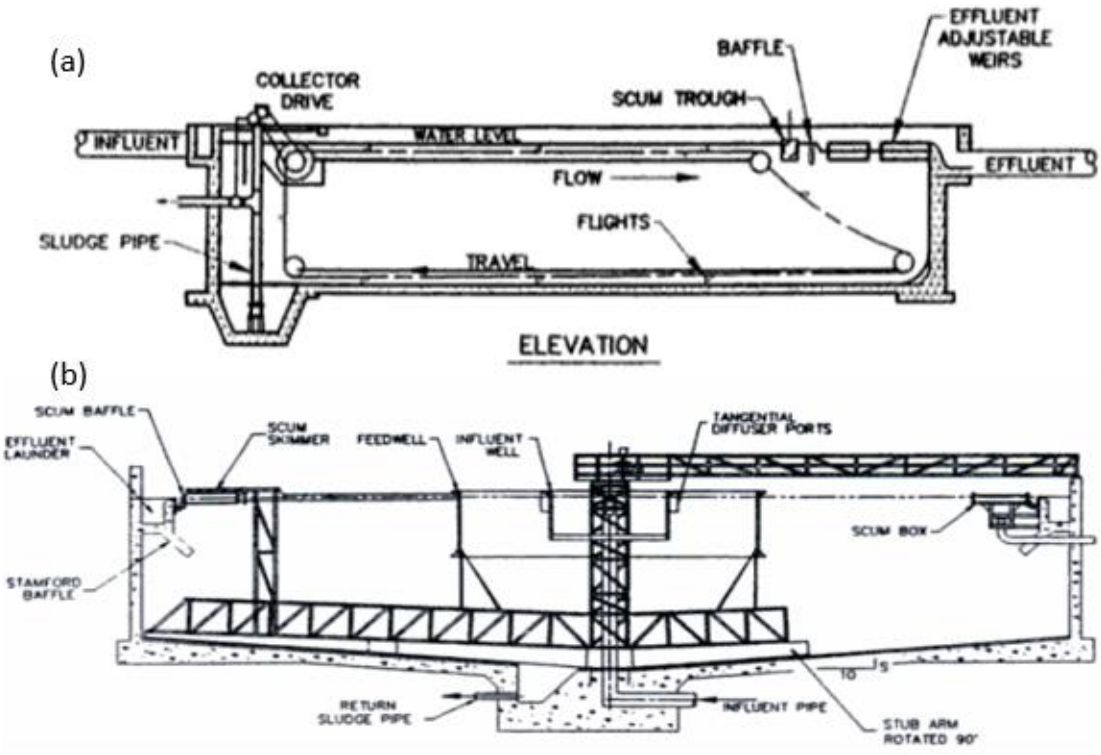

Figure 2-11. Primary clarifiers (a) Rectangular, (b) Circular clarifier [Randall et al., 1992]

2.3.2 Rotating Belt Filter (RBF)

A rotating belt filter (RBF) uses a filter-mesh mounted on an inclined rotating belt to sieve solids from wastewater as shown in Fig. 2-12. As the wastewater flows into the inlet and the belt rotates, the suspended solids are retained on the mesh and the filtered water is conveyed by gravity to the effluent outlet [Franchi and Santoro, 2015]. The performance of the RBF depends on the particle size distribution in the influent wastewater as well as the mesh pore size, and flow rate [Lema and Martinez, 2017]. The pore size of the mesh can typically range from 50 μm to 4000 μm [Ng, 2012], however, 350 μm is the most widely used pore size in full-scale municipal wastewater applications [Paulsrud et al., 2014; Rusten et al., 2017]. RBFs can achieve BOD and TSS removal of 20% and 50%, respectively [Franchi and Santoro, 2015]. The belt speed and water level can be adjusted based on the flow rate to dictate performance. A “filter mat” can be formed that leads to separation of particles smaller than the mesh opening, which enhances the TSS removal efficiency (up to 90%). The “air knife” cleaning feature uses

compressed air to clean the mesh and offers effective sludge thickening (TS 2-8%) as the solids are collected in the solids compartment. A screw press further dewaters the sludge (15%-30% solids) [EPA, 2013; Salsnes Filter, 2015; Franchi and Santoro, 2015; Lema and Martinez, 2017]. Furthermore, although cellulose fibers (as part of the sbCOD), originating from toilet-paper use, have been observed in RBF sludges, no accurate quantitative measurement of cellulose content in RBF sludge has yet been reported [Ruiken et al., 2013; Paulsrud et al., 2014].

The footprint of the RBF unit is approximately one-tenth that of primary clarifiers and the capital cost is 30%-50% less than that of primary clarifiers [EPA, 2013; Franchi and Santoro, 2015]. Additionally, due to high solids concentration, the sludge handling and disposal costs are significantly reduced [Lema and Martinez, 2017]. Besides primary solids separation, RBFs have been proven promising when employed downstream of a moving bed biofilm reactor (MBBR) [Ng, 2012], as well as for harvesting microalgae [Barragan, 2013]. There are a number of pilot (Canada, USA, etc.) and full-scale (Netherlands, Norway, USA, Denmark) installations of the RBF worldwide for primary treatment of wastewater [Franchi and Santoro, 2015; Jansen, 2016; Rusten et al., 2017]. In this thesis, the commercially available RBF called *Salsnes Filter* (Trojan Technologies, London, Canada) was studied.

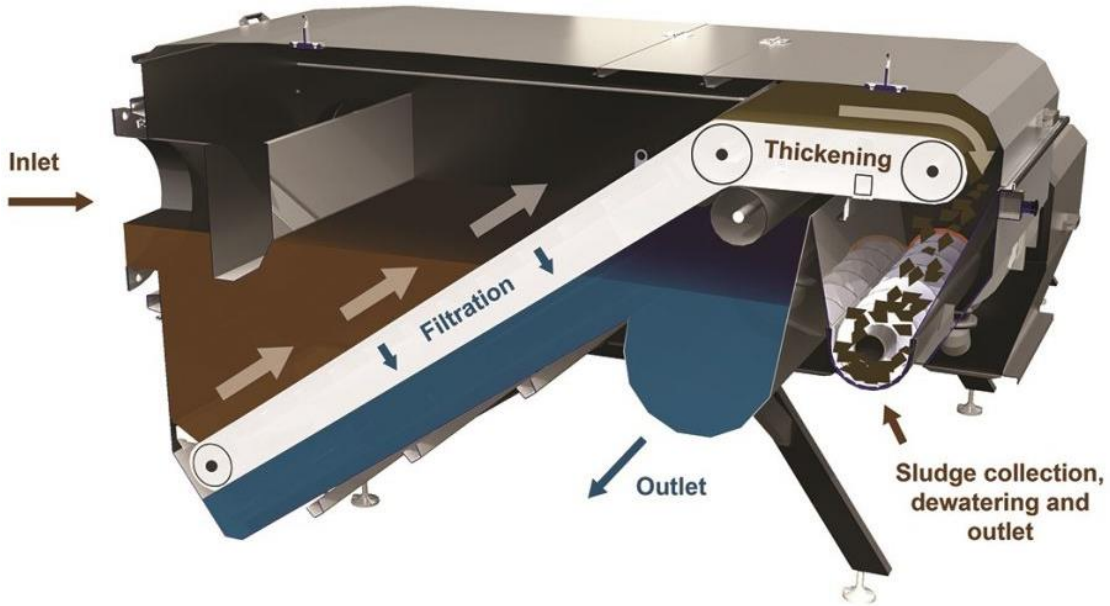

Figure 2-12. Schematic of a Rotating Belt Filter

2.3.3 Ballasted Flocculation Systems

2.3.3.1 Actiflo® Process

Actiflo® is a high-rate chemical clarification process where microsand is used as a ballast particle followed by rapid sedimentation using Lamella plates. Coagulants are added in the coagulation tank to destabilize the suspended solids followed by an injection tank where the microsand is added. The microsand provides a large surface area for the suspended solids to bind causing them to settle rapidly in the clarifier. The collected solids are pumped to a hydrocyclone which separates the sand from the sludge (Fig. 2-13). The high-rate settling and shorter retention times result in smaller footprint than conventional clarifiers [EPA, 2013, Veolia, 2012]. Actiflo® is an established process with installations worldwide for the treatment of surface water, groundwater, wet weather flows, as well as, primary, tertiary, CSO, and industrial applications [Blumenschein et al., 2006; CH2M Hill, 2007]. TSS concentrations of <20 mg/L (90% TSS removal) have been reported during wet weather flows [Veolia, 2012; CH2M Hill, 2007].

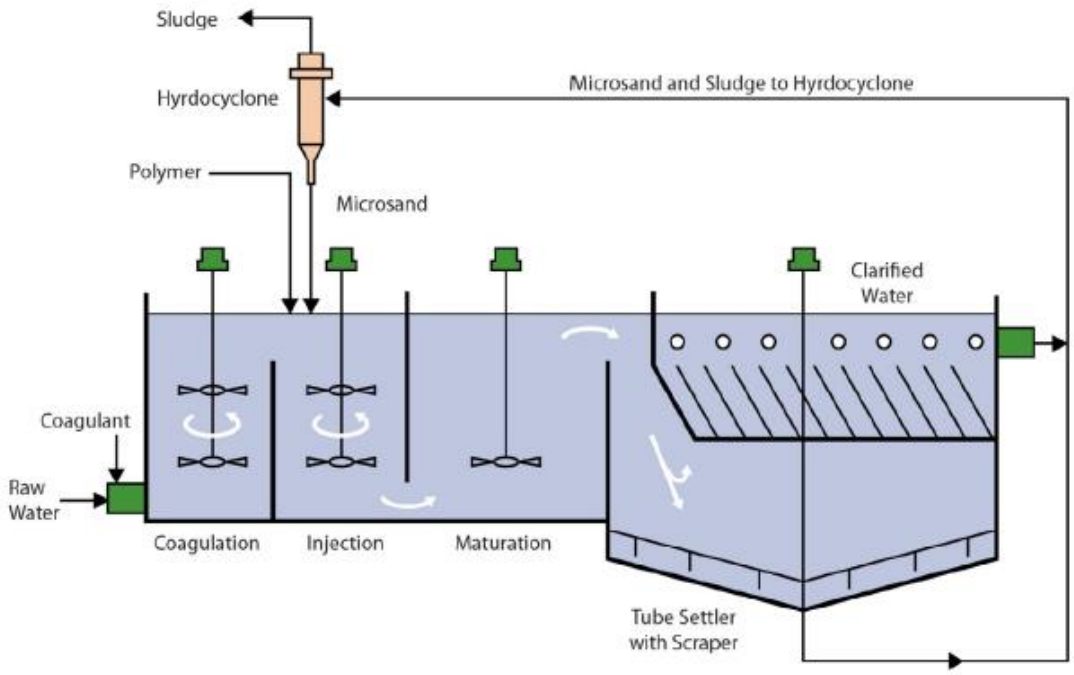

Figure 2-13. Actiflow® process flow diagram [EPA, 2013]

2.3.3.2 DensaDeg® Process

DensaDeg®, similar to Actiflo®, uses sludge as a ballast particle. Coagulant is added in the first stage which overflows into a reactor where sludge and polymer are added (Fig. 2.14). The sludge allows the suspended particles to bind and form high-density flocs which settle rapidly in a Lamella clarifier. Settled sludge gets progressively thickened and recycled back to the reactor zone and excess sludge is wasted [Infilco, 2011]. DensaDeg® requires a smaller footprint compared to conventional clarifiers [EPA, 2013]. DensaDeg® has installations worldwide for primary wastewater, CSO, and tertiary wastewater applications. DensaDeg® can achieve 85% TSS removal efficiency [CH2M Hill, 2007].

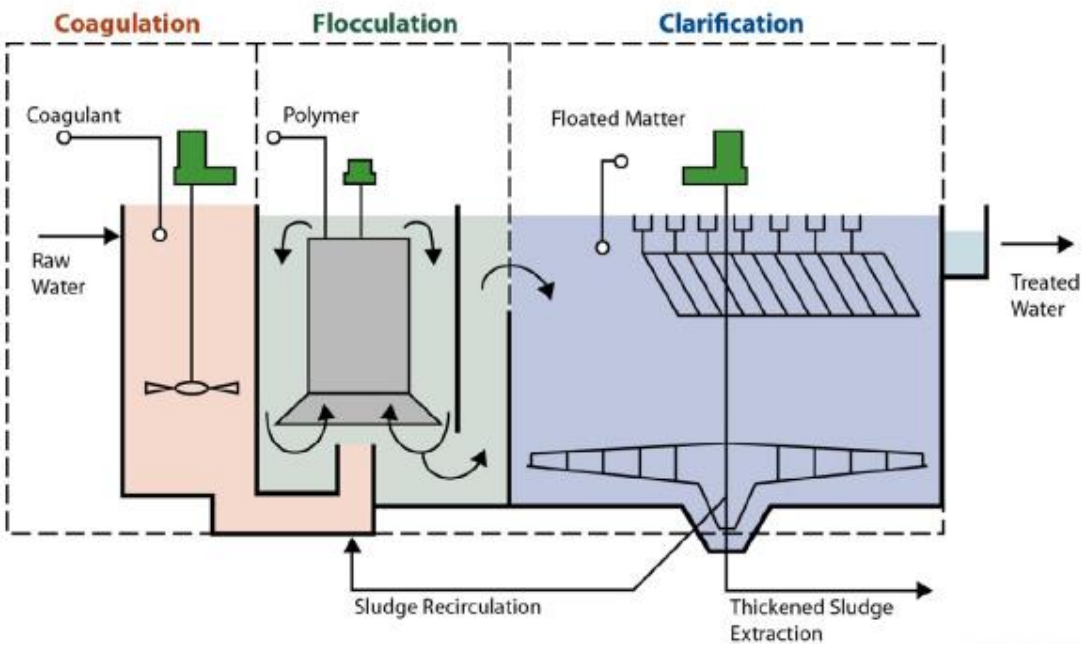

Figure 2-14. DensaDeg® process flow diagram [EPA, 2013]

2.3.4 Critical Review of the Current Primary Technologies

CEPT, Actiflo®, and DensaDeg® are similar technologies that require coagulation, flocculation, and settling. Actiflow® and DensaDeg® are high-rate ballasted clarification processes with performance highly dependent on coagulants, polymers, and ballasting agents. Actiflow® uses microsand, whereas DensaDeg uses sludge as the ballast particle. The ballasting agents generate high-density flocs which are removed by settling. RBF on the other hand, is independent of coagulant and polymer addition for its operation, although, chemically-enhanced RBF is currently being evaluated (including this thesis) for its viability in mainstream treatment [Rusten et al., 2017]. Solids separation occurs continuously as the wastewater flows through the inclined rotating belt filter.

Table 2-7 illustrates an overall comparison between conventional clarifiers, CEPT, RBF, Actiflo® and DensaDeg®. Actiflo® and DensaDeg® can achieve high TSS and BOD removal compared to conventional primary clarifiers. Compared to other technologies, RBF achieves comparable or lower (TSS and BOD) removal than primary clarifiers depending on the particle size distribution. Actiflo® and DensaDeg® have low hydraulic retention times and high overflow rates due to faster settling of flocs [Blumenschein et

al., 2006]. Typically, overflow rates, used in conventional primary clarifiers range between 2-5 $m^3/m^2.h$ which results in detention times of 90-150 min. RBF occupies approximately $1/10^{th}$ of the space requirement of a conventional clarifier [Franchi and Santoro, 2015]. Similarly, Actiflo® and DensaDeg® have small footprint requirements. Due to the smaller footprint requirements, RBF, Actiflo® and DensaDeg® have low capital costs compared to conventional clarifiers, especially where land acquisition is expensive [EPA, 2003]. RBF offers 30%-60% lower capital costs compared to conventional clarifiers. Additionally, RBF has lower operational costs and lower lifecycle costs [Salsnes Filter, 2015]. The major advantage of reduced surface area of clarifiers in Actiflo® and DensaDeg®, minimizes short-circuiting and flow patterns caused by wind and freezing. Ballasted flocculation can treat a wide range of flows without compromising performance [EPA, 2003]. Similarly, compared to the primary clarifiers, RBF are not subjected to short-circuiting due to thermal stratification, wind, and high flow rates and biological activity within the sludge blanket at the bottom of the clarifier [Franchi and Santoro, 2015].

Table 2-7. Comparison between different primary solids separation technologies [CH2M Hill, 2007; Franchi and Santoro, 2015; Lema and Martinez, 2017]

	Conventional primary clarifier	CEPT with conventional clarifier	RBF	Actiflo®	DensaDeg®
TSS removal, %	50-70	70-80	30-50	74-92	81-90
BOD removal, %	25-40	40-60	20	36-62	37-63
HRT, min	90 to 150	60	-	4 to 7	22
Overflow rate, $m^3/m^2.h$	2-5	2-5	17 to 70	60-200	75-100
Chemical addition	No	Yes (20-60 g/m^3 coagulant + 0.5-2.0 g/m^3 polymer)	Optional (0.5-4.0 g/m^3 polymer)	Yes (40-80 g/m^3 coagulant + 0.5-1.5 g/m^3 polymer)	Yes (60-120 g/m^3 coagulant + 1.5-2.5 g/m^3 polymer)
Sludge concentration, mg/L	10,000-25,000	20,000-30,000	20,000-200,000	10,000-15,000	25,000-40,000
Startup time, min	-		-	30	15 to 30
Relative footprint	Large	Large	Small 90% less of clarifiers	Small 50% less of clarifiers	Small 50% less of clarifiers
Capital cost	High	High	Medium	High	High
Operational cost	Low		Low	High	High
kWh per kg of TSS removed	0.05-0.09	0.02-0.06	0.15-0.20	0.07-0.20	0.07-0.20
Maintenance	Low	Low	Medium	High	High
Response to dynamic flow conditions	Negative		Positive	Positive	Positive
State of development	Established	Established	Adaptive use (>500 installations)	Adaptive use (>300 installation)	Adaptive use (>200 installations)

2.4 Biological Nitrogen Removal

As mentioned in Sec 2.1.3, influent municipal wastewater nitrogen load is mainly in the form of TKN, of which 60% is ammonia and the remainder is ON (both SON and PON). PON undergoes hydrolysis to form SON. The SON undergoes biodegradation (deamination) to release ammonia via the process of ammonification. This is an important step in wastewater treatment processes because ON cannot be oxidized by nitrifying bacteria. Ammonia is also the form in which the bacteria incorporate nitrogen for growth [Henze et al., 2008]. An overview of the nitrogen cycle is depicted in Fig. 2-15 [Andalib, 2011]. Nitrogen removal in the biological activated sludge process is achieved by two processes: nitrification and denitrification [Gerardi, 2002].

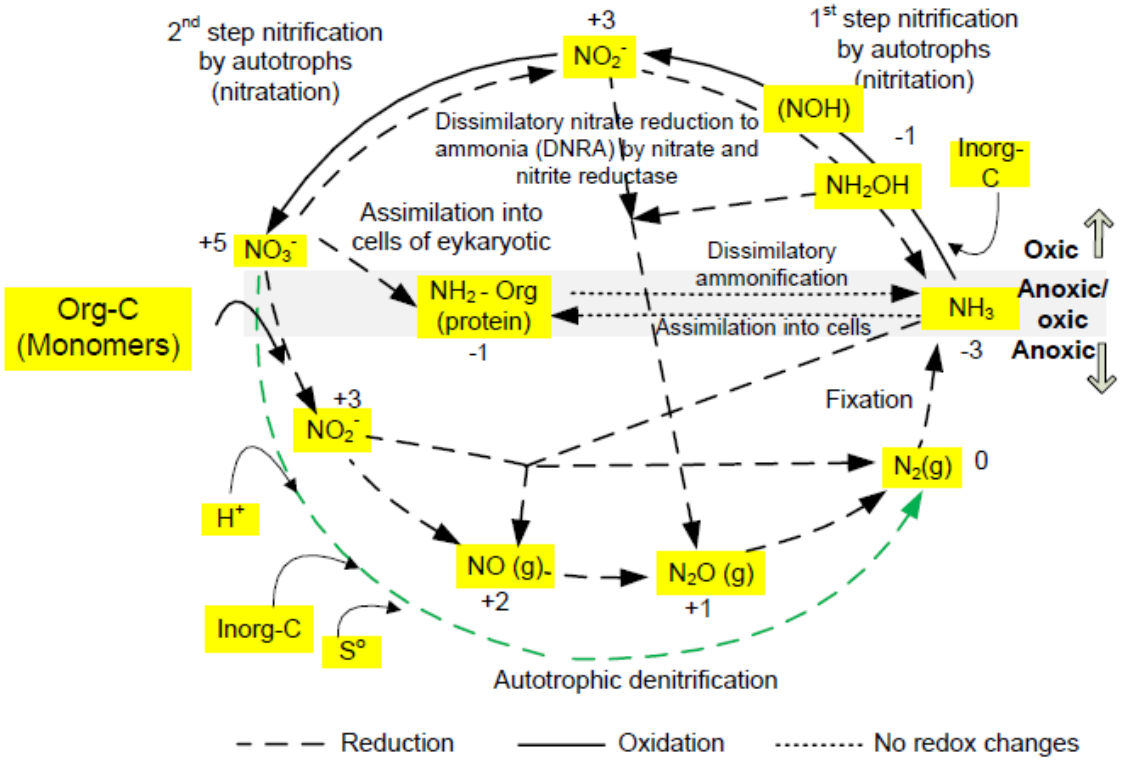

Figure 2-15. Biological nitrogen cycle [Andalib, 2011]

2.4.1 Nitrification

Nitrification is a two-step biological process employing two groups of autotrophic bacteria. The first step, nitritation, involves the oxidation of ammonia to nitrite (NO_2^-) by

ammonium-oxidizing bacteria (AOB), also called *Nitroso*-bacteria (Eq. 2.11). The second step, nitratation, involves the further oxidation of nitrite to nitrate (NO_3^-) by nitrite-oxidizing bacteria (NOB), also called *Nitro*-bacteria (Eq. 2.12) [Tchobanoglous et al., 2003; Rittman and McCarty, 2001].

$$NH_4^+ + 1.5O_2 \rightarrow NO_2^- + 2H^+ + H_2O \quad (2.11)$$

$$NO_2^- + 0.5O_2 \rightarrow NO_3^- \quad (2.12)$$

The most common genus of *Nitroso*-bacteria in wastewater is *Nitrosomonas*, although, *Nitrosococcus, Nitrosopira, Nitrosolobus, Nitrosocystis*, and *Nitrosorobrio* can also oxidize ammonia. *Nitrococcus*, *Nitrospira*, and *Nitroeystis* are the common *Nitro*-bacteria with *Nitrobacter* being the most dominant one. Nitrifying bacteria use carbon dioxide (inorganic carbon) in the form of bicarbonate alkalinity as the carbon source for cell synthesis, and additionally a fraction of the ammonium ions is used as a nutrient source and assimilated into new cells, which can be represented in the following Eq. 2.13 [Gerardi, 2002]:

$$4CO_2 + HCO_3^- + NH_4^+ + 4H_2O \rightarrow C_5H_7NO_2 + 5O_2 + 3H_2O \quad (2.13)$$

The overall nitrification reaction for the complete oxidation of ammonia to nitrate is shown in Eq. 2.14:

$$NH_4^+ + 1.863O_2 + 0.098CO_2 \rightarrow 0.0196C_5H_7NO_2 + 0.98NO_3^- + 0.0941H_2O + 1.98H^+ \quad (2.14)$$

Based on Eq. 2.14, for every g of ammonia nitrogen (as N) converted, 4.25 g O_2 is utilized, 0.16 g of new cells are formed, 7.07 g of alkalinity are removed, and 0.08 g of inorganic carbon is utilized for synthesis of new cells [Tchobanoglous et al., 2003].

Approximately 90%-97% of the bacterial population in activated-sludge process consist of heterotrophs and only 3%-10% of the population are nitrifiers [Gerardi, 2002]. Furthermore, due to the more restrictive energy yielding metabolism of nitrifying bacteria, the maximum specific growth rate of nitrifying bacteria is much lower than heterotrophs (Table 2.8), hence requiring much longer solid retention times (SRT) for nitrifying systems, 10-20 d at 10 ^oC and 4-7 d at 20 ^oC [Tchobanoglous et al., 2003]. The

biomass yield of AOBs and NOBs are 0.15 and 0.02 mg cell/NH_4-N oxidized, while the decay rates of AOBs and NOBs are in the range of 0.05-0.4 d^{-1} and 0.09-0.4 d^{-1} [Cervantes, 2009; Grady et al., 2011].

Table 2-8. Comparison between the maximum specific growth rate of AOBs, NOBs, and heterotrophs [Grady et al., 2011]

	AOB	NOB	Aerobic Heterotrophs	Anoxic Heterotrophs
Maximum specific growth rate, d^{-1}	0.4-1.9	0.5-1.0	6	3.1

Temperature, pH, and dissolved oxygen (DO) are important operating parameters for the nitrification process. The effect of temperature on growth rate can be expressed by the following van't Hoff-Arrhenius equation (Eq. 2.15) [Cervantes, 2009]:

$$\mu = \mu_{20}.\theta^{T-20} \quad (2.15)$$

where μ is the rate coefficient (d^{-1}), μ_{20} is the μ at 20°C (d^{-1}), θ is the temperature coefficient (1.123; dimensionless), and T is the temperature (°C). AOB and NOB are not affected similarly, and NOB are more sensitive to variations of the environmental conditions. At elevated temperatures (>15°C), AOBs have a higher growth-rate than NOBs [Zhu et al., 2008; Cervantes, 2009]. The inhibitory effect of cold temperature is greater on NOBs than AOBs [Gerardi, 2002]. Figure 2-16 illustrates the influence of temperature on the growth rate of AOBs and NOBs [Zhu et al., 2008].

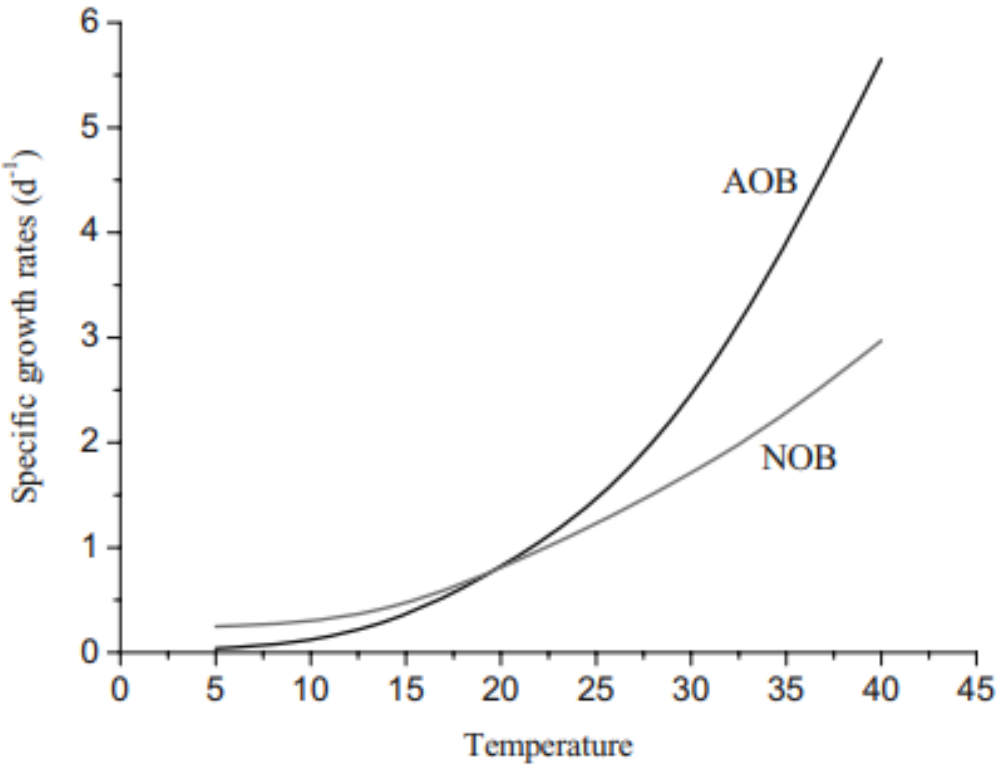

Figure 2-16. Influence of temperature on growth rate of AOBs and NOBs [Zhu et al., 2008]

Nitrification reactions are pH sensitive and can affect the process in two ways: (1) directly by changing the enzyme reaction mechanism and (2) indirectly by changing the ammonium/ammonia (NH_4^+/NH_3) and nitrite/nitrous acid (NO_2^-/HNO_2) equilibrium. Free ammonia (FA) and free nitrous acid (FNA) are inhibitory to nitrification [Cervantes, 2009; Park et al., 2010]. Optimal nitrification occurs at neutral to moderately alkaline conditions (pH 7.5-8 range) [Gerardi, 2002; Tchobanoglous et al., 2003; Cervantes, 2009]. Similarly, nitrification is very sensitive to low DO concentrations due to high half-saturation constant for oxygen. Nitrification rates increase up to DO concentrations of 3 to 4 mg/L. Due to the various inhibitory substances, a wide range of nitrification rates have been reported, 0.25-0.77 g NH_4-N/g VSS.d at 20 ^oC [Andalib, 2011]. Eq. 2.16 accounts for the effect of DO on the specific growth rate of nitrifying bacteria:

$$\mu_n = \left(\frac{\mu_{nm}N}{K_n+N}\right)\left(\frac{DO}{K_o+DO}\right) - k_{dn} \qquad (2.16)$$

where μ_n=specific growth rate of nitrifying bacteria (d^{-1}), μ_{nm}=maximum specific growth rate of nitrifying bacteria (d^{-1}), N= nitrogen concentration (mg/L), K_n=substrate concentration at one-half the maximum specific substrate utilization rate (0.06-5.6 mg/L as NH_4-N for AOBs and 0.06-8.4 mg/L as NO_2-N for NOBs), K_o=half-saturation concentration for DO (0.2-0.4 mg/L for AOBs and 1.2-1.5 mg/L for NOBs), and K_{dn}=decay coefficient for nitrifying bacteria (d^{-1}). Nitrification is inhibited at DO concentration <0.5 mg/L, particularly for NOBs [Tchobanoglous et al., 2003]. Overall, in all cases, NOBs are more sensitive to inhibition than AOBs.

Based on the mass balance over a completely mixed reactor system and the Monod kinetics, the theoretical minimum sludge age for nitrification was derived to be (Eq. 2.17) [Henze et al., 2008]:

$$SRT_{min} = \frac{1}{\mu_{nm,T} - k_{dn}} \qquad (2.17)$$

2.4.2 Denitrification

The biological reduction of nitrates or nitrites to nitric oxide, nitrous oxide, and nitrogen gas (mainly) by facultative heterotrophs to degrade carbonaceous BOD (cBOD) is termed denitrification [Tchobanoglous et al., 2003; Gerardi, 2002]. Denitrification causes dissimilatory nitrogen removal because the nitrate and nitrite ions are reduced to molecular nitrogen, and not assimilated into cellular matter [Gerardi, 2002].

The majority of denitrifying organisms are facultative heterotrophic bacteria that can utilize nitrate or nitrites when oxygen is limiting (anoxic; ORP range of +50 to -50 millivolts) [Gerardi, 2002], and the largest number of denitrifying bacteria are in *Alcaligenes*, *Bacillus*, and *Pseudomonas* genera [Gerardi, 2002]. Some denitrifiers (*Bacillus* and *Chromobacterium*) can perform fermentation in the absence of nitrate or oxygen [Gerardi, 2002; Tchobanoglous et al., 2003]. There is another group of denitrifying organisms (*Thiobacillus denitrificans* and *Thiomicrospira denitrificans*) that are autotrophic denitrifiers that use reduced sulfur compounds as electron donors while using nitrate as the electron acceptor and carbon dioxide as the carbon source; therefore, achieving desulfurization and denitrification simultaneously [Zou et al., 2016].

The stepwise reduction of nitrate involves the sequential conversion of nitrate to nitrite, nitric oxide (NO), nitrous oxide (N_2O), and molecular nitrogen (Eq. 2.18):

$$NO_3^- \rightarrow NO_2^- \rightarrow NO \rightarrow N_2O \rightarrow N_2 \quad (2.18)$$

Biological oxidation of cBOD using nitrate or nitrite can be expressed in two biochemical reactions (Eq. 2.19 and 2.20) as follows:

$$NO_3^- + cBOD \rightarrow NO_2^- + CO_2 + H_2O \quad (2.19)$$

$$NO_2^- + cBOD \rightarrow N_2 + CO_2 + H_2O \quad (2.20)$$

cBOD is the electron donor for nitrate removal and the availability of cBOD needed for nitrate removal in denitrification process is an important design parameter. Sources of cBOD in denitrification process includes the bsCOD in the wastewater, bsCOD produced during endogenous decay, and external source such as methanol, ethanol or acetate. The following reactions shows the nitrate removal using different sources of cBOD (Eq. 2.21, 2.22, and 2.23):

$$Wastewater: C_{10}H_{19}O_3N + 10NO_3^- \rightarrow 5N_2 + 10CO_2 + 3H_2O + NH_3 + 10OH^- \quad (2.21)$$

$$Methanol: 5CH_3OH + 6NO_3^- \rightarrow 3N_2 + 5CO_2 + 7H_2O + 6OH^- \quad (2.22)$$

$$Acetate: 5CH_3COOH + 8NO_3^- \rightarrow 4N_2 + 10CO_2 + 6H_2O + 8OH^- \quad (2.23)$$

For every g of nitrate reduced, one equivalent alkalinity is produced, that is, 3.57 g alkalinity (as $CaCO_3$) production per g NO_3-N reduced. Thus, in denitrification half the alkalinity lost in nitrification, can be recovered [Tchobanoglous et al., 2003]. cBOD is also used for the synthesis of the new biomass as shown in Eq. 2.24 (with methanol as the carbon source) [WEF, 2005a]:

$$NO_3^- + 1.08CH_3OH + 0.24H_2CO_3 \rightarrow 0.04C_5H_7NO_2 + 0.48N_2 + 1.23H_2O + HCO_3^- \quad (2.24)$$

It has been estimated that 4 g BOD_5 is removed for every g of NO_3 denitrified, although it is dependent on the type of carbon source used and operating conditions. The actual amount can be calculated using Eq. 2.25 [Tchobanoglous et al., 2003]:

$$\frac{g\ bsCOD}{g\ NO_3{-}N} = \frac{2.86}{1-1.42Y_n} \quad (2.25)$$

where Y_n is the net biomass yield, g VSS/g bsCOD.

A wide range of COD/N ratios between 4 and 15 g COD/g N have been reported in the literature for complete denitrification [Peng et al., 2007].

Denitrification processes are typically designed using the specific denitrification rate (SDNR). Three distinct denitrification rates have been observed in predenitrification (preanoxic) tanks using cBOD in influent wastewater: the first rate associated with rapid (fast) denitrification using rbCOD; the second rate associated with slow denitrification with particulate and colloidal COD; and third rate associated with endogenous respiration (very slow) [Peng et al., 2007; WEF, 2005a]. The first SDNR ranges from 0.07 to 0.32 g NO_3-N/g VSS.d, the second rate of 0.08 gNO_3-N/g VSS.d, while the third-rate ranges from 0.04 to 0.05 g NO_3-N/g VSS.d [Razafimanantsoa et al., 2014a]. SDNR depends on the concentration of active biomass, rbCOD in the influent, and temperature. However, the following empirical relationship (Eq. 2.26) can be used to get a conservative estimate of SDNR in predenitrification system [WEF, 2005a, Tchobanoglous et al., 2003]:

$$SDNR = 0.03\left(\frac{F}{M}\right) + 0.029 \quad (2.26)$$

where, SDNR is the specific denitrification rate (g NO_3-N/g MLVSS.d); and F/M is the g BOD applied per g MLVSS per day in the anoxic tank.

Denitrification rate in postanoxic system can be determined using Eq. 2.27 [WEF, 2005a]:

$$SDNR = 0.12\ SRT^{-0.706} \quad (2.27)$$

where SRT is the solids retention time (days).

Denitrification rates are proportional to the substrate utilization rate. DO can be inhibitory to nitrate reduction enzymes, therefore, the denitrification rates can be expressed by the following Eq. 2.28:

$$r_{su} = -\left(\frac{kXS}{K_S+S}\right)\left(\frac{NO_3}{K_{S,NO_3}+NO_3}\right)\left(\frac{K_O}{K_O+DO}\right)\eta \quad (2.28)$$

where η is the fraction of denitrifying bacteria in the biomass; K_O is the DO inhibition coefficient for nitrate reduction; $K_{S,NO3}$ is the half velocity coefficient for nitrate limited reaction.

Table 2-9 summarizes the literature studies using various carbon sources for denitrification. These carbon sources can be categorized as: 1) pure chemicals such as methanol, ethanol, acetate etc.; 2) purified agricultural or industrial byproducts; 3) raw industrial/agricultural byproducts such as corn syrup, other process wastes; 4) sludge fermentation products and 5) others such as methane. Due to low cost, favorable kinetics, and low cell yield, methanol has been an industrial standard [Onnis-Hayden and Gu, 2008]. There is a wide range for the denitrification rates for methanol, ranging from 0.05-0.32 g N/g VSS.d. Comparing other pure chemicals, ethanol, acetate, propionate, butyrate, and lactate produce high removal rates than methanol or glucose. Several studies have explored industrial/agricultural waste products to enhance denitrification and have reported favorable kinetics. However, there are some drawbacks associated with these wastes including the availability and consistency of these wastes; additionally, pretreatment of the waste is usually required [Onnis-Hayden and Gu, 2008]. Sludge based (digester supernatant) carbon sources have also been studied to avoid the cost of external carbon and the denitrification rates are similar to rates obtained with acetate [Bilanovic et al., 1999]. Different hydrolysis methods have been used including biological, chemical, and physical, to improve the bioavailability of carbon from the sludge which are comparable to rates obtained with acetate [Onnis-Hayden and Gu, 2008].

Table 2-9. Summary of literature using various carbon sources for denitrification

Carbon source	COD/ NO_3-N	Overall rate g N/g VSS.d	Reference
Methanol		0.208-0.323	Beccari et al., 1983
		0.072	Nyberg et al., 1996
	3.7	0.28	Carrera et al., 2003
		0.077	Peng et al., 2007
		0.228	Dold et al., 2008
		0.055	Onnis-Hayden and Gu, 2008
	4.8	0.146	Onnis-Hayden and Gu, 2008
	6	0.151	Dholam et al., 2014
		0.156	Chen et al., 2015
Ethanol	20	0.043	Gerber et al., 1986
		0.24	Nyberg et al., 1996
		0.230	Peng et al., 2007
		0.134	Onnis-Hayden and Gu, 2008
		0.204	Dold et al., 2008
		0.156	Chen et al., 2015
Methanol + Ethanol		0.180	Dold et al., 2008
Acetate		0.24-0.48	Henze et al., 1994
	2-9	0.076-0.175	Naidoo et al., 1998
	2.04	0.475	Bilanovic et al., 1999
		0.288	Peng et al., 2007
	5.7	0.326	Onnis-Hayden and Gu, 2008
		0.204	Chen et al., 2015
	6	0.380	Zhang et al., 2016
Acetic acid	20	0.060	Gerber et al., 1986
	13	0.667	Akunna et al., 1993
	3-5	0.941	Lee & Welander, 1996
		1.140	Frison et al., 2013
	6	0.152	Li et al., 2015
Lactic acid	20	0.052	Gerber et al., 1986
	15.5	0.667	Akunna et al., 1993
Propionate		0.168	Chen et al., 2015

Table 2-9. Summary of literature using various carbon sources for denitrification (cont.)

Carbon source	COD/ NO_3-N	Overall rate g N/g VSS.d	Reference
Propionic acid	20	0.040	Gerber et al., 1986
	6	0.075	Li et al., 2015
Butyric acid	20	0.051	Gerber et al., 1986
Formic acid	20	0.036	Gerber et al., 1986
Glycerol	14.5	0.178	Akunna et al., 1993
		0.110	Frison et al., 2013
	5	0.312	Chen et al., 2013
Glucose	20	0.022	Gerber et al., 1986
	15	0.065	Akunna et al., 1993
	8.9	0.091	Onnis-Hayden and Gu, 2008
		0.168	Chen et al., 2015
	6	0.072	Zhang et al., 2016
Raw wastewater	2-9	0.079-0.124	Naidoo et al., 1998
	7.67	0.080	Razafimanantsoa et al., 2014a
	3.4-7.5	0.019-0.084	Yan et al., 2017
Raw wastewater + centrifuge	2-9	0.078-0.136	Naidoo et al., 1998
Raw wastewater + coagulated + centrifuge	2-9	0.072-0.130	Naidoo et al., 1998
Anaerobic digester effluent	2.04	0.486	Bilanovic et al., 1999
Hydrolyzed/Fermented sludge		0.118-0.180	Onnis-Hayden and Gu, 2008
Hydrolyzed/Fermented sludge post alkalinization		0.146-0.182	Onnis-Hayden and Gu, 2008
Fermented sludge	7.2	0.234	Moustafa, 2004
Alkaline hydrolysed sludge	7.2	0.134	Moustafa, 2004
Thermal hydrolyzed sludge	6.9	0.286-0.382	Onnis-Hayden and Gu, 2008
Sewage + methanol	4.5	0.141	Dholam et al., 2014

Table 2-9. Summary of literature using various carbon sources for denitrification (cont.)

Carbon source	COD/ NO_3-N	Overall rate g N/g VSS.d	Reference
Fermented municipal solid waste	1.6-2.4	0.12	Bolzonella et al., 2001
Organic fraction of municipal solid waste fermentation liquid		0.510-0.650	Frison et al., 2013
Cattle manure + maize silage fermentation liquid		1.16	Frison et al., 2013
Starch wastewater		0.018	Peng et al., 2007
Starch wastewater + ethanol		0.051	Peng et al., 2007
Potato processing waste	5	0.288	Chen et al., 2013
Crude syrup	3-5	0.499	Lee & Welander, 1996
Hydrolyzed starch	3-5	0.518	Lee & Welander, 1996
Distillery Fusel Oils	2.22	0.331	Onnis-Hayden and Gu, 2008
Pea blanch water	5.71	0.259	Onnis-Hayden and Gu, 2008
MicroC™	6.4	0.113-0.153	Onnis-Hayden and Gu, 2008
Methane		0.6	Thalasso et al., 1997
Salsnes Filter with 150 µm mesh	6.10	0.10	Razafimanantsoa et al., 2014a
Salsnes Filter with 33-150 µm mesh	9-14	0.048-0.054	Razafimanantsoa et al., 2014b
Food waste fermentation liquid	6	0.309	Zhang et al., 2016

2.5 Nitrogen Removal Systems

Biological nitrogen removal processes include cBOD removal with nitrification and denitrification. There are sequential processes with alternating environments to ultimately achieve total nitrogen removal. In order to denitrify, nitrification must be completed, at least partially [WEF, 2005a]. Aerobic zones oxidize the organic matter to CO_2, H_2O, and new heterotrophic biomass is generated. In addition, NH_4-N is oxidized to NO_3-N along with growth of nitrifying bacteria. Anoxic zones allow the NO_3-N formed by nitrifying bacteria to be converted to nitrogen gas by denitrifying bacteria, thereby achieving nitrogen removal from wastewater. The most common suspended growth process configurations are described in Table 2-10 [Tchobanoglous et al., 2003; WEF, 2005a; Zhu et al., 2008].

Table 2-10. Description of suspended growth processes for nitrogen removal

Process	Description
Wuhrmann Process	The Wuhrmann process configuration (Fig. 2-17), is a single-sludge post-anoxic system. Due to lack of cBOD available in the anoxic zone, denitrification is proportional to the endogenous respiration rate in the mixed liquor, therefore, long detention times are required in the post-anoxic tank.
Ludzack-Ettinger	The Ludzack-Ettinger (1962) configuration (Fig. 2-18), is a pre-anoxic system which takes advantage of the cBOD in the influent wastewater. The process depends on the nitrates returning in the RAS, therefore, denitrification is limited by the RAS recycle ratio. The advantages of this configuration include alkalinity production before nitrification as well as BOD removal before aerobic zone saves aeration energy.
Modified Ludzack-Ettinger (MLE)	MLE is one of the most widely used BNR processes. The original Ludzack-Ettinger process (Fig. 2-19) was improved by providing internal recycle of the mixed liquor from the aerobic zone to the anoxic zone. Both the denitrification rate and the overall nitrogen removal efficiency are improved in this configuration. 5 to 8 mg/L of TN is achievable. Typical internal recycle rates ranges from 2 to 4. One of the key challenges of the MLE process is the dissolved oxygen in the recycled mixed liquor, and for this reason DO control is required before recycle. The recycle ratio is dependent on the dissolved oxygen and the oxygen demand of influent wastewater (primarily rbCOD). SRT ranges from 7-20 day.

Table 2-10. Description of suspended growth processes for nitrogen removal (cont.)

Process	Description
4-Stage Bardenpho	The four-stage Bardenpho (Fig. 2-20) incorporates the MLE (pre-anoxic) and the Wuhrmann (post-anoxic) processes to include two anoxic zones to achieve high total nitrogen removal. The first two stages work as an MLE process, however the first anoxic zone is sized larger to accommodate mixed liquor recycle rate. The majority of the denitrification occurs in the first anoxic tank and the portion of flow that is not recycled back is denitrified in the second anoxic tank. The second aerobic tank strips any nitrogen gas formed in the second anoxic tank and increase dissolved oxygen concentration before secondary clarification to improve sludge settling. TN of <3 mg/L can be achieved in this configuration. Typical SRT ranges from 10-20 day.
Sequential Batch Reactor (SBR)	The SBR (Fig. 2-21) is a fill-draw system where all the processes are conducted in a single reactor following a sequence of fill, reaction, settling, and decant phases in a cycle. Typically, SBRs complete 4-6 cycles per day for domestic wastewater and 50% to 75% of the liquid volume is retained at the end of every cycle. Wastewater is added in the 'Fill' phase, raising the liquid level and mixing is commenced. The 'React' phase can employ an anoxic phase for pre-denitrification followed by an aeration period. In the 'Settle' phase aeration and mixing is stopped, and the biomass is allowed to settle. The 'Decant' phase removes the clarified effluent and the biomass is wasted as necessary. The SBR cycles can be configured to operate as a nitrification system (single or multi-phased aeration), an MLE process, or a Bardenpho system (for both nitrogen and phosphorous removal). The SBR process is very flexible, however, operation requires automation and operator attention. 8 mg/L of TN is achievable and typical SRT ranges from 10-30 day.
A2O Process	The anaerobic/anoxic/aerobic (A2O) process (Fig. 2-22) is a modification to the MLE configuration with an anaerobic zone before the anoxic. The A2O system achieves both nitrogen and phosphorous removal. Nitrate rich mixed liquor from the end of aerobic zone is returned to the anoxic zone which minimizes the nitrates returned in the RAS to the anaerobic zone. The phosphorous is removed in the anoxic/aerobic zones. One of the limitation of the A2O process is that the nitrates returned in the RAS can affect phosphorous removal performance. Typical SRT ranges from 5-25 day.

Table 2-10. Description of suspended growth processes for nitrogen removal (cont.)

Process	Description
UCT (University of Cape Town)	The UCT process (Fig. 2-23) is a modification to the A2O process in order to minimize the affects of nitrates returned to the anaerobic zone. The RAS is recycled to the anoxic zone instead of the anaerobic zone, and the internal recycle is from the anoxic to the anaerobic zone for increased organic uptake in the anaerobic zone. The UCT is a more complex operation due to an additional recycle stream, however, it achieves improved nitrogen and phosphorous removal. Typical SRT ranges from 10-25 day.

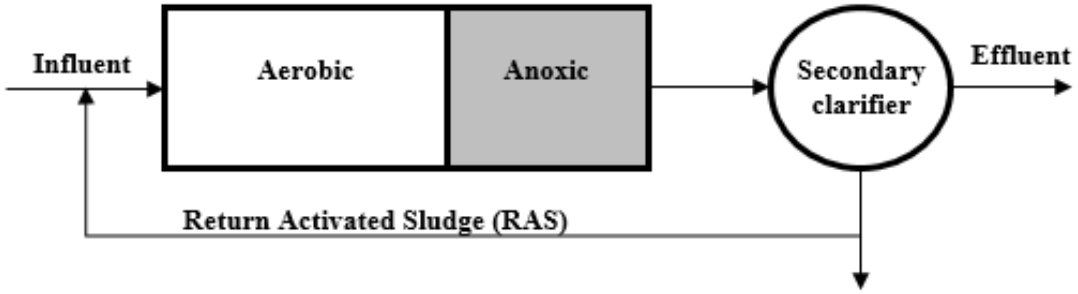

Figure 2-17. Wuhrmann Process configuration for nitrogen removal

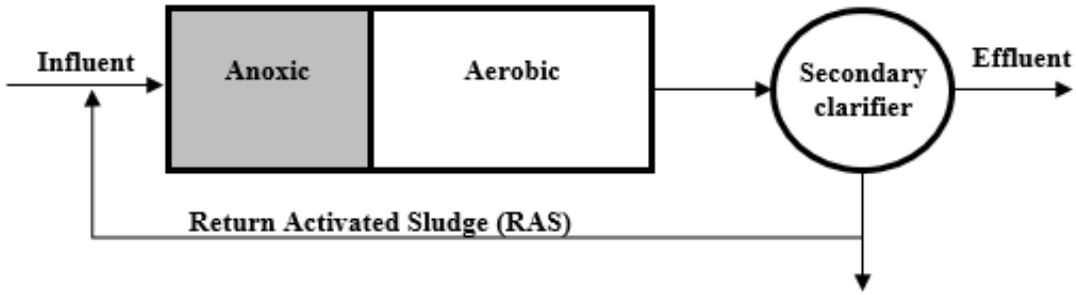

Figure 2-18. Ludzack-Ettinger process configuration for nitrogen removal

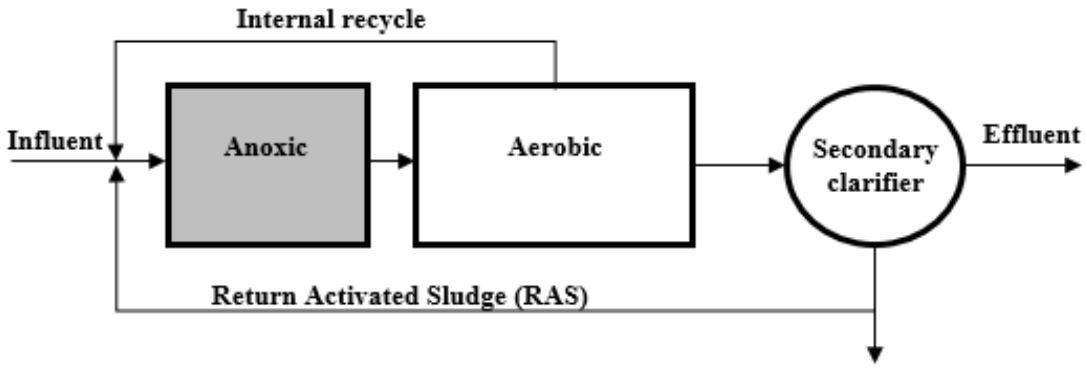

Figure 2-19. Modified Ludzack-Ettinger (MLE) process configuration for nitrogen removal

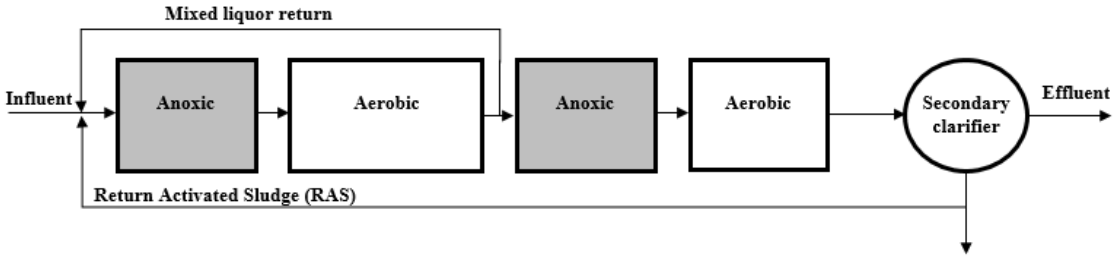

Figure 2-20. 4-Stage Bardenpho configuration for nitrogen removal

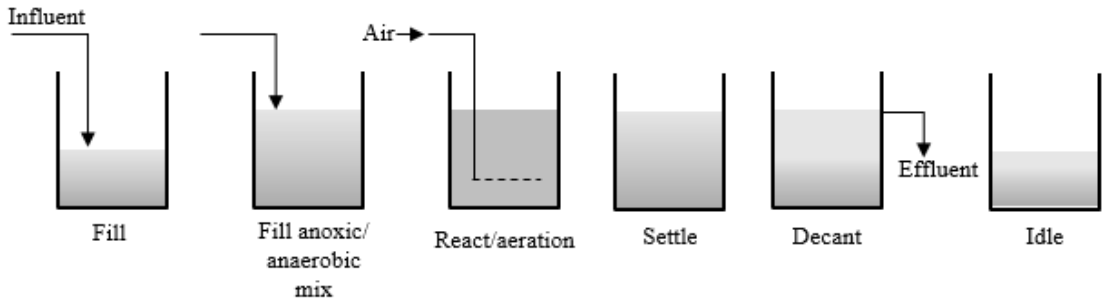

Figure 2-21. Sequential Batch Reactor (SBR) process cycles for nitrogen removal

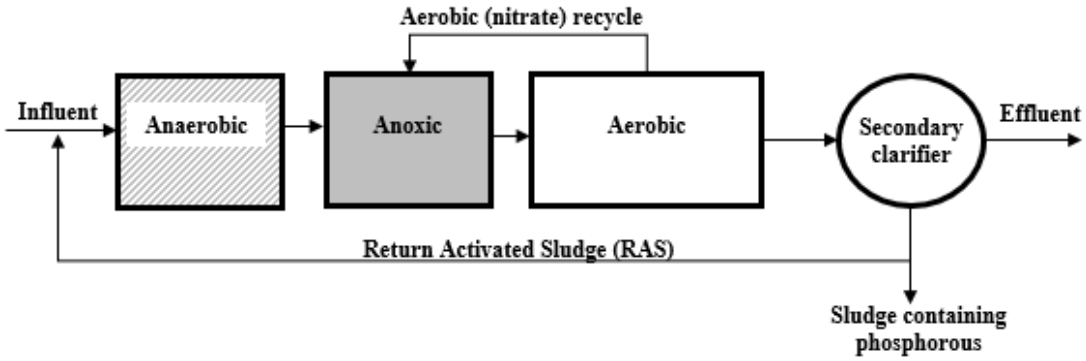

Figure 2-22. Anaerobic/anoxic/aerobic (A2O) process configuration for nitrogen and phosphorous removal

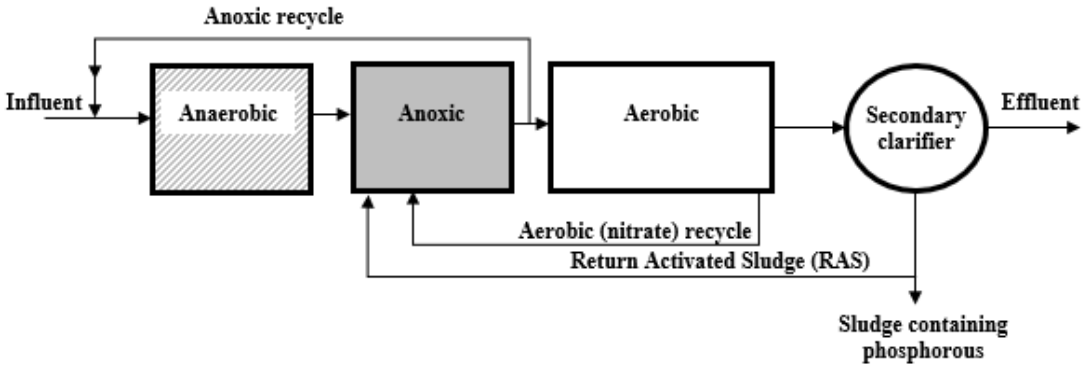

Figure 2-23. UCT (University of Cape Town) process configuration for nitrogen and phosphorous removal

2.6 Synopsis of the Literature

Due to ecological needs and progressing eutrophication in receiving waters, regulators have reduced effluent nutrient concentrations to protect the environment. These implementations have led to a shift from traditional physical-chemical processes for nutrient removal to advanced biological nutrient removal to meet more stringent target effluent quality, which for TN is <1.5-3 mg/L and TP is <0.07 mg/L. These limits are driving the research towards combination of more efficient solids separation technologies

and enhanced BNR by retrofitting existing treatment facilities [EPA, 2008; Oleszkiewicz and Barnard, 2006].

Primary clarification is the most widely used primary process for solids separation with many installations and is a well understood process. Although primary clarifiers are easy to operate and offer reliable removal performance (TSS: 50-70% and BOD: 25-40%), there are certain drawbacks of this technology. Under varying flow conditions, primary clarifiers do not offer consistent removal. The static nature of primary clarifiers causes unintentional fermentation due to the formation of a sludge blanket which alters the characteristics of the waste sludge which affects downstream sludge handling processes including anaerobic digestion. Additionally, primary clarifiers have a large footprint and have relatively high capital costs.

Actiflo® and DensaDeg® both offer small footprint and similar TSS (85-95%) and BOD removal (50-60%) but such aggressive solids removal, although reduces aeration demand and biological sludge production, is not beneficial for denitrification and biological phosphorous removal processes as they require carbon to drive nutrient removal.

Salsnes Filter, a rotating belt filter technology, has been labelled as an ‘emerging’ technology by EPA [2013] for removal of primary solids. TSS and BOD removal of ~30-50% and 20%, respectively, can be achieved using Salsnes Filter. The RBF has several advantages over primary clarifiers. Since solids are separated based on particle size distribution, the RBF can be optimized to target specific fractions in the wastewater. RBF is a continuous process which offers consistent effluent and sludge quality [Sarathy et al., 2015]. Additionally, it requires the $1/10^{th}$ the footprint of a primary clarifier and has an integrated sludge thickening and dewatering mechanism.

There has been increased interest in carbon diversion technologies for improved carbon management to fit in the scheme of wastewater resource recovery facilities. Thus, it is imperative to evaluate the role of RBFs as a primary solids-separation technology in wastewater resource recovery facilities.

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Chapter 3

3 Experimental Assessment and Validation of Quantification Methods for Cellulose Content in Municipal Wastewater and Sludge

3.1 Introduction

The wastewater treatment industry is evolving from the traditional goals of effective control of environmental and health impacts of wastewater discharge to increased sustainability and decreasing costs by minimizing energy costs and resource recovery [Ruiken et al., 2013]. Typically, organic matter in wastewater is characterized by surrogate parameters like chemical oxygen demand (COD), total organic carbon (TOC), and biochemical oxygen demand (BOD) and the main organic contaminants have been identified as protein, carbohydrates, and lipids [Raunkjær et al., 1994]. Of the insoluble pollutants in wastewater treatment plant influents, cellulose, in the form of toilet paper, has been reported to be a major component which inadvertently ends up in sewage sludge [Edberg and Hofsten, 1975; Verachtert et al., 1982]. Toilet paper consumption in North America amounts to around 1.9 kg per capita per month [Ruiken et al., 2013]. Based on 400 L wastewater produced per person per day, 220 mg total-suspended-solids (TSS) per L wastewater, and the above-mentioned statistics on toilet paper consumption, wastewater can contain up to 158 mg toilet paper/L, that is, about 72% of the TSS. The determination of cellulose in wastewater is thus indispensable to understand its fate in wastewater treatment facilities as well as its recovery potential.

Cellulose is the most abundant organic polymer on earth and is intimately associated with numerous aspects of human advancements including fuel, shelter, clothing, food, and paper [Bauer and Ibanez, 2014; Harris et al., 2010; Olsson and Westman, 2013; Thoorens et al., 2014]. Cellulose is considered a complex carbohydrate very similar to starch, and is a linear polymer of β-1,4-glycosidic bond linked with β-D-glucose units [Olsson and Westman, 2013; Rinaldi and Schüth, 2009; Thoorens et al., 2014]. The degree of polymerization (DP), which is directly related to solubility, is the number of glucose units in a cellulose chain. Lack of branching and unique conformation of hydroxyl groups

causes chains of cellulose to form, and the dense intramolecular hydrogen bonds provide chain stiffness forming crystalline structures that are insoluble in water and most of the common solvents [Bauer and Ibanez, 2014; Rinaldi and Schüth, 2009]. Of the three classes of cellulose, α-cellulose is the pure form of cellulose with high (greater than 200) DP whereas β-cellulose (DP less than 30) and γ-cellulose (DP 50-200) are associated with the hemicellulose constituent of plant material [Bolam, 1965]. Microcrystalline cellulose, also known as Avicel (brand name derived from the original company name – American Viscose Cellulose), is a partially depolymerized α-cellulose, prepared by treating α-cellulose with mineral acids [Thoorens et al., 2014].

Cellulose is a valuable resource which if recovered can be used for various other applications such as production of fuels and chemicals, building materials, bioplastics, flocculants etc. [Pellizzer, 2016; Rinaldi and Schüth, 2009]. Accordingly, when it is recovered, sludge disposal costs could be reduced substantially [Faust et al., 2014; Honda et al., 2002] and oxygen consumption and concomitant energy use for biodegradation are eliminated. To this end, new processes and technologies have been developed and validated at full scale such as the one based on the Cellvation™ concept, recently developed through a number of Horizon 2020 European projects (http://www.cirtec.nl/en/gebruikt-toiletpapier-krijgt-tweede-leven/). This process, based on the use of the microsieving technology (e.g., Salsnes Filter), has shown significant potential for cellulose recovery from raw wastewater with potential downstream increase in biological processing capacity due to the removal of COD. Moreover, due to the low extent of cellulose biodegradability in the aeration tank, the removal of cellulose and other fiber-like material is expected to lead to additional operational savings such as lower aeration energy consumption and secondary sludge production.

However, in order to investigate the fate of cellulose during wastewater treatment, lack of accuracy for cellulose determination in wastewaters and sludges must be addressed. Of the different methods studied in the literature, acid-hydrolysis and enzymatic hydrolysis of cellulose are the most widely studied methods. Both methods are based on the principle of hydrolyzing cellulose to monosaccharides, with the glucose yield indicating the cellulose content in the sample.

Table 3-1 summarizes some of the literature studies that explored one-stage and two-stage acid-hydrolysis of cellulose. Updegraff [1969] observed 100% glucose yield using concentrated (72%) sulfuric acid as the hydrolyzing agent. On the other hand, Camacho et al. [1996], also using concentrated (70%) sulfuric acid, observed only 32% glucose yield from microcrystalline cellulose. Gavilla et al. [2015] and Kim et al. [2001] used diluted sulfuric acid for hydrolysis at high temperatures (120 and 205 ^{o}C, respectively) but only achieved about 60% yield of microcrystalline cellulose and α-cellulose, respectively. Orozco et al. [2007] also studied dilute acid hydrolysis of cellulose at higher temperature but by using phosphoric acid at 7.5% acid concentration at 160 ^{o}C and observed 55% yield. As a final one-step hydrolysis method, Chimentao et al. [2014] used oxalic acid at 65 and 120 ^{o}C for a prolonged treatment, and achieved 85% yield.

Yoon et al. [2014] reported 90% yield in microcrystalline cellulose using a two-stage acid hydrolysis method (National Renewable Energy Laboratory, i.e., NREL method). This NREL method was developed to determine the structural carbohydrates and lignin in biomass. The procedure uses a two-step acid hydrolysis to fractionate biomass into easily quantifiable forms [Sluiter et al., 2012]. The first-stage 1-hour hydrolysis uses 72% sulfuric acid that disrupts the crystalline structure of cellulose resulting in release of glucose units. The 1 to 2 hour second-stage hydrolysis utilizes 4% sulfuric acid digestion which yields hemicellulosic sugars i.e., xylose, arabinose, mannose, and galactose [Bauer and Ibanez, 2014; Gao et al., 2014]. The glucose yield of these two stage methods was 90-93% for pure cellulose and microcrystalline cellulose, respectively.

Xiang et al. [2003] described acid-hydrolysis of cellulose as a complex heterogeneous reaction involving hydrolytic chemical reaction factors as well as nonreaction factors impacted by various factors such as state of hydrogen bonding, crystallinity, diffusion barrier, chemical composition, swelling state of cellulose, etc. In addition to the above-mentioned factors, decomposition of hydrolysis products (by dehydration) as a second step following hydrolysis is another challenge [Rinaldi and Schüth, 2009]. Based on the aforementioned studies, it appears that acid hydrolysis is not the most reliable method for cellulose determination.

Table 3-1. Literature review of cellulose determination methods

Cellulose type	Acid	Contact time (h)	Temperature (°C)	Yield (%)	Reference
α-cellulose	72% sulfuric acid	1	Room temperature	100%	[Updegraff, 1969]
Microcrystalline cellulose (Avicel)	70% sulfuric acid	20	40 °C	32%	[Chamacho et al., 1996]
Microcrystalline cellulose (Avicel)	3 % sulfuric acid	4	120 °C in a microwave reactor system	57 %	[Gavila et al., 2015]
α-cellulose	0.07% sulfuric acid	0.5	205 °C	62%	[Kim et al., 2001]
Cellulose (type unknown)	7.5% phosphoric acid	0.08	160 °C in a microwave reactor system	55%	[Orozco et al., 2007]
Microcrystalline cellulose	6% oxalic acid	6	120 °C	85%	[Chimentao et al., 2014]
Microcrystalline cellulose (Avicel) (Two-stage acid hydrolysis)	72% sulfuric acid	1	30 °C	90%	[Yoon et al., 2014]
	4% sulfuric acid	2	100 °C		
Microcrystalline cellulose (Avicel) (Two-stage acid hydrolysis)	72% sulfuric acid	1	Room temperature	93%	[Bauer and Ibanez, 2014]
	4% sulfuric acid	1	121 °C		

Similarly, varying glucose yields have been observed with enzymatic hydrolysis depending on the cellulose source tested. While promising and reliable results are obtained using model cellulosic substrates (like α-cellulose), the results cannot be extrapolated to 'real' samples. A number of substrate-related and enzyme-related effects and their interactions play an important role in the hydrolysis efficiency and are the most challenging aspect of this method [Mansfield et al., 1999; Yang et al., 2011]. For instance, cellulose's structure, crystallinity, DP, and accessible surface area impact enzyme adsorption which directly correlates to hydrolysis yields [Mansfield et al., 1999; Yang et al., 2011]. Similarly, enzyme-related factors, such as thermal instability, products inhibition, enzyme inactivation, have been reported to impact the hydrolysis of cellulose [Yang et al., 2011]. Consequently, numerous studies perform different pre-treatments (such as hydrogen peroxide, potassium hydroxide, sulfuric acid, hydrochloric acid, and HCl/KOH), prior to enzymatic hydrolysis to depolymerize cellulosic fibers into products with low DP which facilitate substrate-enzyme contact [Alkasrawi et al., 2016; Camacho et al., 1996; Champagne and Li, 2009; Rinaldi and Schüth, 2009]. These pre-treatments have been reported to enhance end-product yields from 31% to 69% by facilitating swelling of cellulose that alters the crystalline structure of cellulose, decreases the DP, and expanding the specific surface area for enzyme accessibility.

The majority of the research done on acid and enzymatic hydrolysis treatment has been focused on the industrial hydrolysis of cellulose to glucose and cellodextrins (short-chain cellulose oligomers) with the ultimate goal of producing fuels and chemicals [Rinaldi and Schüth, 2009], and accordingly the reliability and accuracy of cellulose measurement was secondary to final product yield quantification. The Schweitzer method, named after the Swiss chemist Matthias Eduard Schweizer (1818-1860) who invented the Schweizer also called Schweitzer reagent (cuprammonium hydroxide solution) [Kauffman, 1984], developed by Hurwitz et al. [1961] was originally intended to determine cellulose in sewage sludge but despite promising recovery of cellulose and good reproducibility, this method was never further explored in the literature for wastewater-related research. The aforementioned authors focussed only on temporal variation of cellulose measurements in activated sludge to correlate that with an operational problem of fibrous heat-dried activated sludges causing problems with mechanical equipment, with no attempt of

method validation. In the authors opinion, two potential reasons for the lack of further interest in the Schweitzer method for wastewater applications could be that there was no interest in determining cellulose in wastewater before and the issue has only recently garnered attention due to transition in the wastewater treatment industry towards resource recovery. Additionally, the authors also believe that researchers nowadays no longer search into older journal articles that are not readily accessible through internet search engines. Although this reagent did not garner attention in wastewater research, the Schweitzer reagent has been used successfully in experimental botany research [Fuller and Barshad, 1960] as well as to isolate cellulose from soil samples [Gupta and Sowden, 1964]. The most widely used application of the Schweitzer reagent is in the textile industry, i.e. in the production of synthetic cellulose products such as rayon [Seymour and Johnson, 1976]. In contrast to the aforementioned methods, the Schweitzer method does not depend on the hydrolysis to glucose. The Schweitzer reagent is an excellent solvent for cellulose and forms a complex with the cellulose that upon acidification or in alcoholic conditions, precipitates, allowing the cellulose to be measured gravimetrically.

The objective of this work was to compare the different cellulose measurement methods and to determine the most reliable method to accurately quantify cellulose in a complex matrix of wastewater and sludge. A good method should be reproducible, accurate (no bias with actual cellulose content), have fixed recovery (preferably 100%), quick or with little hands-on time, and cheap in terms of chemicals and equipment. Four different methods were tested for the above-mentioned criteria including dilute-acid hydrolysis, concentrated acid hydrolysis, enzymatic hydrolysis, and the Schweitzer method. The underlying principle of the three hydrolytic method is that cellulose is hydrolyzed to glucose.

3.2 Material and Methods

For the determination of cellulose, in this paper four methods were tested, three of which used hydrolysis followed by soluble products determination, and one gravimetric measurement. For the identification of the best method for cellulose determination, the

tests were first performed using α-cellulose (Sigma Aldrich, Ontario, Canada) as a standard to avoid interferences. Thereafter, primary clarifier sludge and sieved primary sludge (sludges arising from sieving raw wastewater through a 350 µm sieve) [Sarathy et al., 2015] samples were used to confirm the performance of the methodology. The sludge samples were collected from the Greenway WWTP, located in London, Ontario, Canada. The average total solids content of primary clarifier sludge and sieved primary sludge was 3±0.01% and 5±0.24%, respectively. The sludge samples were dried at 105 ^{o}C (VWR Gravity Convection Oven, Ontario, Canada) overnight prior to testing.

3.2.1 Acid Hydrolysis

Acid hydrolysis was conducted using 5% sulfuric acid and a cellulose concentration of 20 g/L. An initial test was done where 0.2 g of α-cellulose, toilet paper, and sieved solids were added to 10 mL of 5% sulfuric acid solution in a lightly capped glass vial. The reaction was carried out at 100 ^{o}C. 1 mL samples were taken at predetermined time intervals and the glucose concentration was determined using glucose kits (Biopacific Diagnostics, Ontario, Canada). A second test was done and the reaction volume was increased to 100 mL. The cellulose yield was computed as the measured glucose concentration divided by the cellulose mass added (Eq. 3.1) as follows:

$$Cellulose\ yield\ (\%) = \frac{Glucose\ concentration\left(\frac{g}{L}\right) \times volume\ (L)}{substrate\ added\ (g)} \times 100\ \% \qquad (3.1)$$

3.2.2 Enzymatic Hydrolysis

Enzymatic hydrolysis was conducted following the method of Champagne and Li [2009]. Although Champagne and Li [2009] recommended using 10 % (by weight) cellulase concentration, in this work different cellulase concentrations ranging from 1 % to 20 % cellulase-to-cellulose concentration ratios were tested. The first test was carried out on α-cellulose where the equivalent weight of α-cellulose (2 g, dry mass) and cellulase enzyme corresponding to the respective enzyme loading, were added to 100 mL of sodium citrate buffer (pH 4.8) in a 125-mL batch bottle. The batches were placed in a shaker where the temperature was maintained at 40 ^{o}C and shaken (Thermo Scientific MaxQ4000 Shaker, Ontario, Canada) at 160 rpm. Samples were withdrawn at predetermined time intervals

and the glucose concentrations were determined using glucose kits. Equation 1 was used to calculate the percentage cellulose yield. The method was also tested on sieved primary sludge samples.

3.2.3 NREL Method

As a third alternative, the NREL method was tested to measure for its potential to measure cellulose in wastewater and sludge. This method uses a two-step acid hydrolysis to hydrolyze the sludge into soluble forms that can be quantified using HPLC [Sluiter et al., 2012]. In the first step, 0.3 g of sample (dry mass) was added to a glass vial and 3 mL of 72 % sulfuric acid was added. The mixture was stirred using a glass tube and placed in a water bath set at 30 ^{o}C for 1 h. After 1 h incubation, the tubes were removed from the water bath and diluted to 4 % sulfuric acid by adding 84 mL of deionized water. The samples were thoroughly mixed and placed in an autoclave at 121 ^{o}C in the liquid setting for 1 h. After autoclaving, the samples were allowed to cool to near room temperature. The samples were filtered through a 0.45 µm filter paper and 20 mL of filtrate was collected in a 50-mL vial. Calcium carbonate was used to neutralize the sample to pH 5-6. The neutralized samples were subsequently filtered through a 0.2 µm syringe filter and analyzed for glucose, cellobiose, xylose, galactose, arabinose, and mannose using an HPLC (Hewlett Packard Model 1090 HPLC with a refractive index detector; HPLC column: BioRad Aminex7 HPX-87C). In order to assess if the method could differentiate between cellulose and starch, an initial test was also conducted with different cellulose: starch mass ratios including 0:1, 1:3, 1:1, 3:1, and 1:0. The method was also tested on toilet paper and sieved sludge samples.

3.2.4 Schweitzer Method

Cellulose forms a soluble complex with the Schweitzer reagent but precipitates in an alcohol solution [Hurwitz et al., 1961]. The Schweitzer reagent was prepared by adding 5.5 g of cupric hydroxide to 1 L of 28 % to 29 % ammonium hydroxide and the mixture was stirred for 30 min. The reagent has a deep blue colour. The following procedure was applied to determine the cellulose content using the Schweitzer method. First, the samples were pretreated to remove protein and other impurities. 0.1 to 0.3 g of sample

(dry mass) was added to an Erlenmeyer flask and diluted to 200 mL with distilled water. To this sample 1.25 mL of 50 % NaOH solution and 5 mL antifoaming agent (Sigma Aldrich, Ontario, Canada; diluted in proportion of 1 part defoamer to 5 parts water) was added. The mixture was boiled for 30 min. The mixture was then cooled and 300 mL of distilled water was added. The diluted mixture was transferred to a centrifuge bottle and a centrifugal force of 724 $x\ g$ was applied for 20 min (Beckman Coulter Allegra 6 Centrifuge). The supernatant was decanted, and the pellet was washed with 300 mL of distilled water and centrifuged again. The supernatant was discarded, and 100 mL of the Schweitzer reagent was added to the pellet. The pellet was broken using a spatula and the bottles were placed in a mechanical shaker for 60-90 min at 120 rpm. The bottles were centrifuged, and the supernatant was collected into another centrifuge bottle containing 300 mL of 80 % ethyl alcohol. The mixture was stirred and allowed to stand for 30 min. After 30 min, the bottles were centrifuged, and the supernatant was discarded. The pellet was washed with 1.25 % HCl (breaking up the pellet using a spatula) until the blue copper colour of the precipitate disappeared completely. The solution was filtered on pre-washed and weighed 1.2 µm glass fiber filters (VWR, Ontario, Canada). The precipitate was washed with distilled water, followed by 10-20 mL of 80 % ethyl alcohol. The filters were dried in a 105 ^{o}C oven overnight and weighed. The filters were ignited in a muffle furnace (Lindberg Blue Box Furnace) at 550 ^{o}C for 60 min and weighed again. The percent cellulose in the sample was calculated using the following equation (Eq. 3.2):

$$\%\ cellulose = \frac{wt.dried\ residue - wt.ignited\ residue}{wt.of\ sample} \times 100 \qquad (3.2)$$

3.3 Results and Discussion

3.3.1 Acid Hydrolysis

Acid hydrolysis is the most widely used method for hydrolysing carbohydrates. In an initial test, different cellulose sources were tested in triplicates including α-cellulose and toilet paper at 20 g/L (dry mass) in 10 mL reaction volume. As can be seen from Fig. 3-1a, the replicates were not reproducible. The highest yield of 50 % was observed for

toilet paper and α-cellulose samples, after 45 h of hydrolysis. It is noteworthy that cellulose yields for two α-cellulose samples were 50% and 42%, and for the three toilet paper samples were 50%, 25%, and 23%.

The reaction volume in the above test was too small and therefore the test was repeated in 100 mL reaction volume at 20 g/L α-cellulose concentration (Fig. 3-1b). The results obtained in this test i.e., the 25 % cellulose yield was much lower than the 50 % yield observed in the initial test and was not very encouraging due to the lack of reproducibility. Several studies have reported overall cellulose yields of 50 % - 60 % at higher temperatures of >200 ^{o}C in typical batch reactors [Kim et al., 2001; Wyman et al., 2005]. Nevertheless, pyrolysis and other side reactions occur at higher temperatures, leading to charring or caramelization of glucose [Orozco et al., 2007; Wyman et al., 2005]. A black residue was indeed observed in this study which evidently may explain the low cellulose yields. There is abundant literature (Table 3-1) that has studied acid hydrolysis using various acids (sulfuric acid, hydrochloric acid, oxalic acid, acetic acid-water-nitric acid, phosphoric acid, etc.) at varying temperatures and conditions, and every study achieved different cellulose yields [Bauer and Ibanez, 2014; Chimentao et al., 2014; Kim et al., 2001; Orozco et al., 2007; Schell et al., 2003; Yoon et al., 2014]. The majority of the research done on dilute-acid treatment has been conducted to hydrolyse cellulose to glucose and cellodextrins (short-chain cellulose oligomers) [Olsson and Westman, 2013]. However, since the objective of this study was to quantify cellulose itself, the inability to duplicate the results of the test does not make this method reliable, and therefore it is not suitable for determining cellulose concentrations.

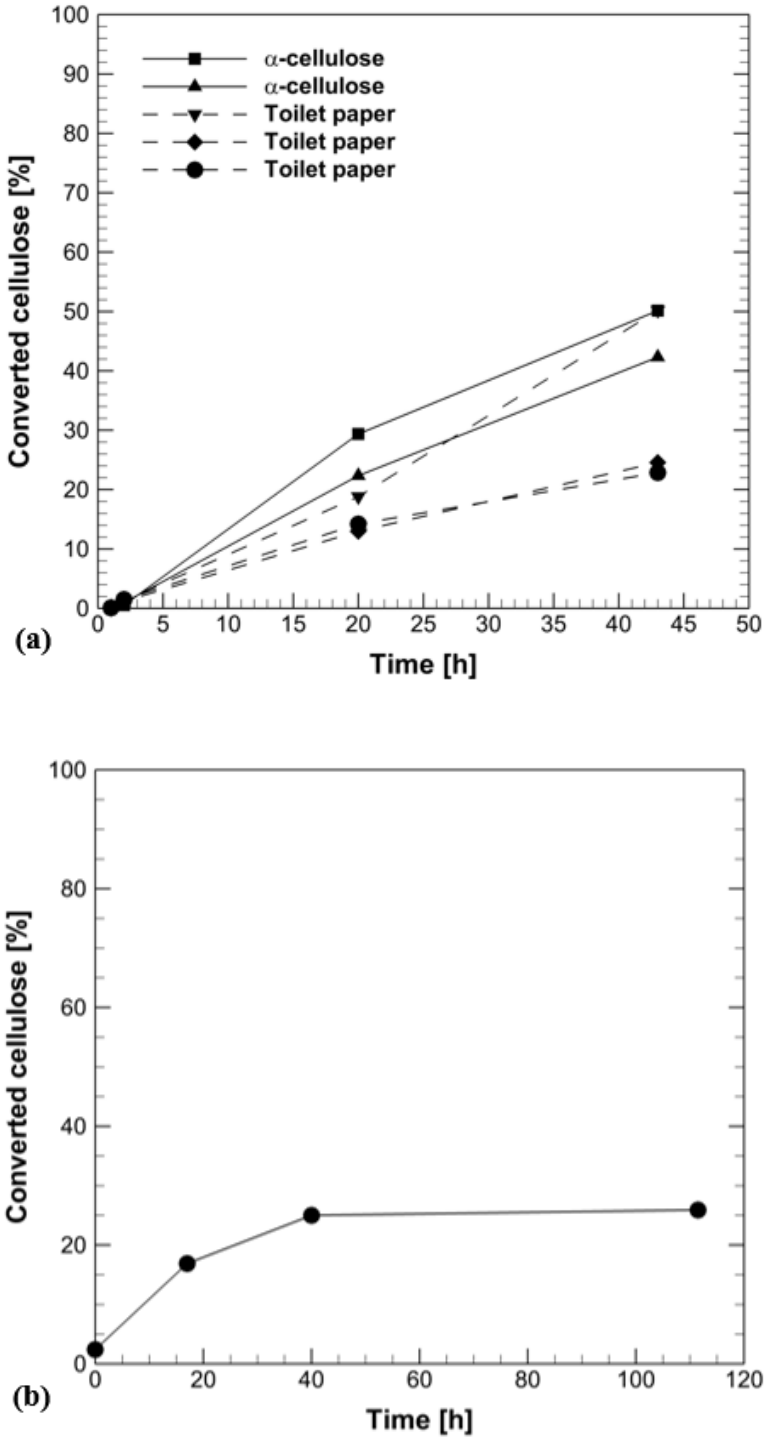

Figure 3-1. Acid hydrolysis method at 100 °C (a) using different cellulose sources in 10 mL reaction volume; (b) at 20 g/L α-cellulose in 100 mL reaction volume

3.3.2 Enzymatic Hydrolysis

Enzymatic hydrolysis is the other widely studied method for cellulose hydrolysis. Although Champagne and Li [2009] recommended using 10 % cellulase-to-cellulose concentration (on a mass basis), in this study different cellulase concentrations ranging from 1 % to 20 % (Fig. 3-2a) were tested. It can be observed from Fig. 3-2a that although the 20 % cellulase condition had the highest rate of cellulose conversion; the yield plateaued at 46 % after 2 d. The highest yield of 67 % cellulose was achieved by the 10 % cellulase.

In order to develop a standard calibration curve for cellulose, the 20 % cellulase dose was selected due to its high rate and another experiment was run using different cellulose concentrations as shown in Fig. 3-2b. We see a similar trend in this test, with the yield plateauing at 47±3 % after 2 d. The test was terminated after 7 d.

The standard curve was plotted at different time intervals and a good linear relation was observed between the cellulose concentration and the measured glucose concentrations with R^2 >0.99 (Fig. 3-3), but the slope of the linear relation was different at different times which makes it extremely difficult to standardise.

Hereafter, the enzymatic hydrolysis method was tested on sieved primary sludge samples and 20% cellulase dose (Fig. 3-4). The aforementioned standard curves (Fig. 3-3a,b) at 1 d and 2 d were used to estimate the cellulose concentrations at different concentrations of sieved primary sludge. Table 3-2 tabulates these results which highlights the inconsistencies in % cellulose estimated in the same sample of sieved primary sludge at different concentrations. Unlike the experiment above that tested α-cellulose, varying yields (ranging from 40% to 83%) were observed at different concentrations of sieved primary sludge (Table 3-2). It is interesting to observe that the higher the concentration of sieved primary sludge, higher the glucose yield (Fig. 3-4).

Theoretically, the specific surface area available for enzyme activity should not be different, however, higher recoveries maybe an artifact of biomass activity and hydrolysis of other carbohydrates to glucose. Champagne and Li [2009] conducted a similar study

where enzymatic hydrolysis of dried primary sludge (4% TS) was performed, and 25 ± 0.8 % conversion was reported after 24 h. This conversion efficiency increased to 37 ± 1 % when the primary sludge was pretreated with both HCl and KOH [Champagne and Li, 2009]. Champagne and Li [2009] also emphasized that the differences in the percentage conversion were due to the cellulose fibers in the sludge being inaccessible to the enzyme due to the complex matrix of the primary sludge, and therefore, pre-treatment with HCl-KOH prior to enzymatic hydrolysis helped isolate cellulosic content from non-cellulosic constituents.

Thus, although enzymatic hydrolysis showed good reproducibility while testing with α-cellulose (Fig. 3-2b), it was not effective with sieved primary sludge samples due to its complex composition. Additionally, Mansfield et al. [1999] emphasized that the results obtained using "purer" model cellulosic substrates cannot be extrapolated to "real' substrates. The efficacy of enzymes in hydrolyzing substrates is intimately linked to the structural characteristics of the substrate such as DP, crystallinity, fiber size, accessible surface area, and the extent of fibrillation [Mansfield et al., 1999].

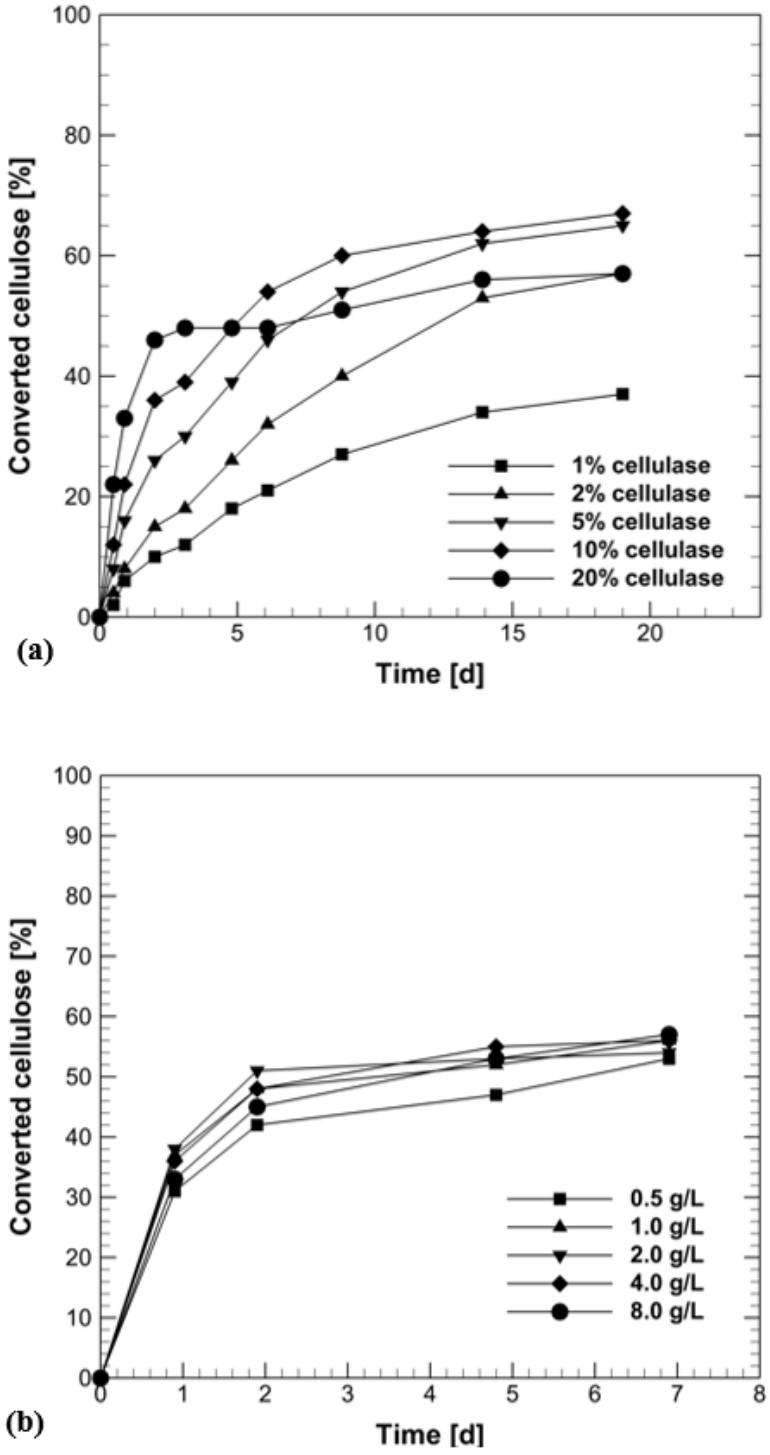

Figure 3-2. Enzymatic hydrolysis (a) at different cellulase dose and 20 g/L α-cellulose; (b) with 20% cellulase dose at different α-cellulose concentrations

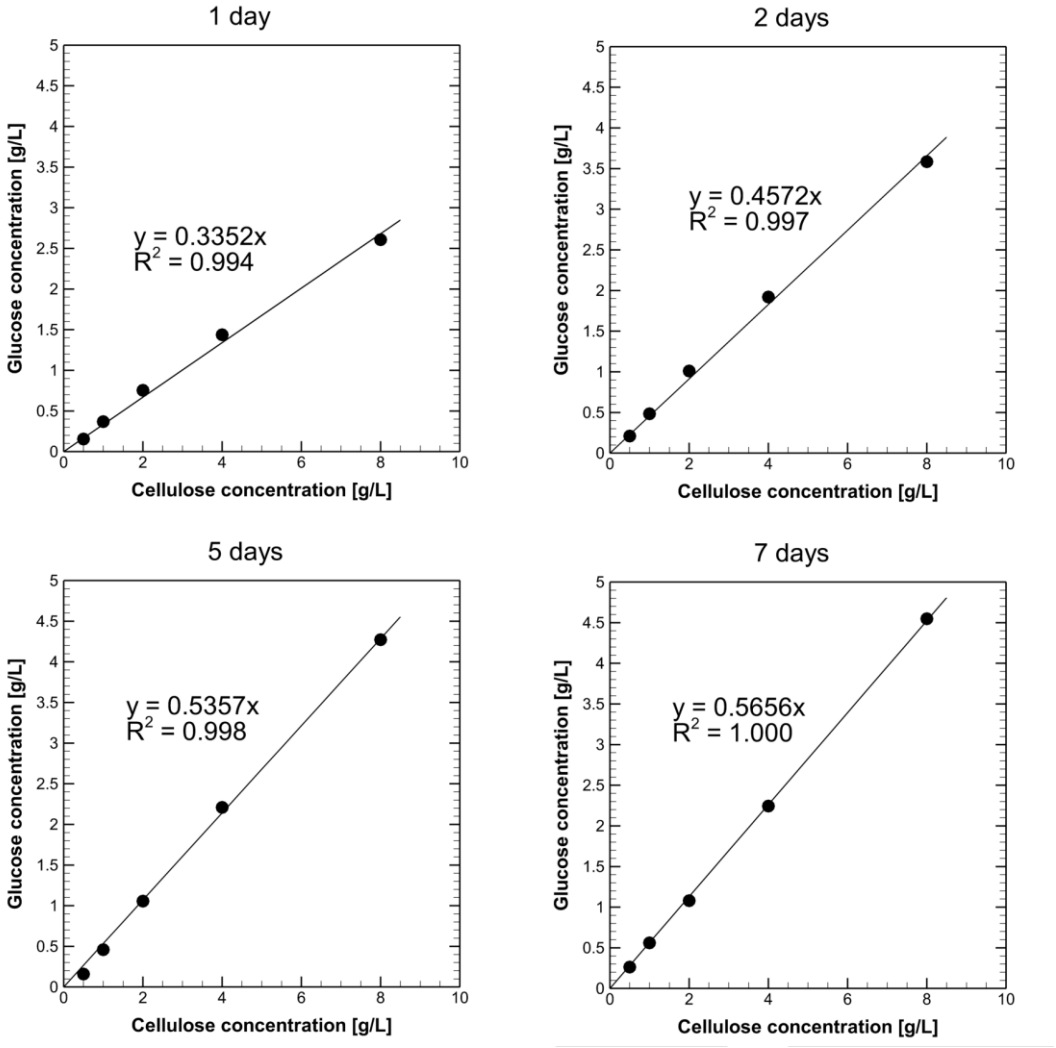

Figure 3-3. Enzymatic hydrolysis; Standard curves at different time intervals: (a) 1 day; (b) 2 day; (c) 5 day; (d) 7 day

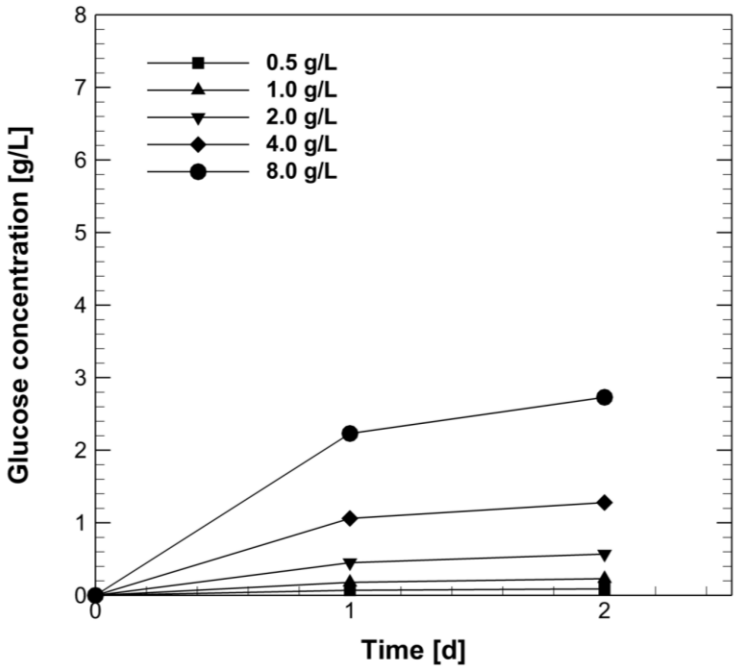

Figure 3-4. Enzymatic hydrolysis with 20% cellulase dose at different sieved primary sludge concentrations

Table 3-2. Estimated % cellulose of sieved primary sludge

	Sieved sludge concentration dosed				
	0.5 g/L	1 g/L	2 g/L	4 g/L	8 g/L
Glucose conc. (g/L) after 1 d	0.07	0.18	0.45	1.06	2.23
Corresponding cellulose conc. (g/L) using Fig. 3-3a standard curve	0.2	0.5	1.3	3.2	6.7
Estimated % cellulose	40	54	67	79	83
Glucose conc. (g/L) after 2 d	0.09	0.23	0.57	1.28	2.73
Corresponding cellulose conc. (g/L) using Fig. 3-3b standard curve	0.2	0.5	1.3	2.8	6.0
Estimated % cellulose	41	50	63	70	75

3.3.3 NREL Method

The NREL method was another method that was tested to measure cellulose. In order to assess whether the method could differentiate between cellulose and starch, an initial test was conducted with different cellulose-to-starch mass ratios including 0:1, 1:3, 1:1, 3:1, and 1:0. Fig. 3-5a shows the mass fraction of soluble sugars to the sum of cellulose and starch added and it is observed that glucose was the predominant sugar detected in all the tests irrespective of the applied cellulose-to-starch mass ratio. The inability to differentiate cellulose from other carbohydrates is the biggest drawback of this method since the aggressive acidic hydrolysis solubilizes both cellulose and starch to glucose.

In order to dismiss this method as a reliable method for cellulose measurement, the NREL test was performed on toilet paper and sieved primary sludge samples with the results depicted in Fig. 3-5b which shows the mass fraction of cellobiose and arabinose relative to the mass of dry sieved primary solids added. Cellobiose (C6) and glucose (C6) are soluble products of cellulose whereas arabinose (C5) is a soluble product of hemicellulose. The sieved primary sludge showed 44±2 % cellobiose as compared to the toilet paper which showed 24±3 % cellobiose. No glucose was detected in either sample; however, a significant amount of arabinose was detected in toilet paper (70±1 %) and sieved primary sludge (38±2 %). However, the reported 70 % hemicellulose content in toilet paper seems to be unrealistically high. Few online sources (http://en.fenjie.com/news/show_223.html (accessed April 28, 2017); http://www.perinijournal.it/Items/en-US/Articoli/PJL-34/New-strength-additive-for-tissue-offers-much-promise (accessed April 28, 2017)) indicate the addition of hemicellulose to cellulose pulp in the making of toilet paper but the precise composition of toilet paper is not available to the best of the authors' knowledge. Alternatively, the authors speculate that perhaps it is not arabinose that is detected but another degradation product. Yoon et al. [2014] studied different second hydrolysis reaction temperatures and observed lower cellulose to glucose conversion at higher temperature of 120 ^{o}C (~70 %) but higher conversion of cellulose to formic acid due to further degradation of glucose in acidic medium to 5-hydroxymethylfurfural (HMF) and then to formic acid and levulinic acid. Similarly, the aforementioned authors also studied combinations of cellulose, xylan,

and lignin, and observed different conversion efficiencies compared to cellulose-alone. The same argument made regarding the effect of structural characteristics on enzymatic hydrolysis applies to the two-stage acid hydrolysis [Mansfield et al., 1999].

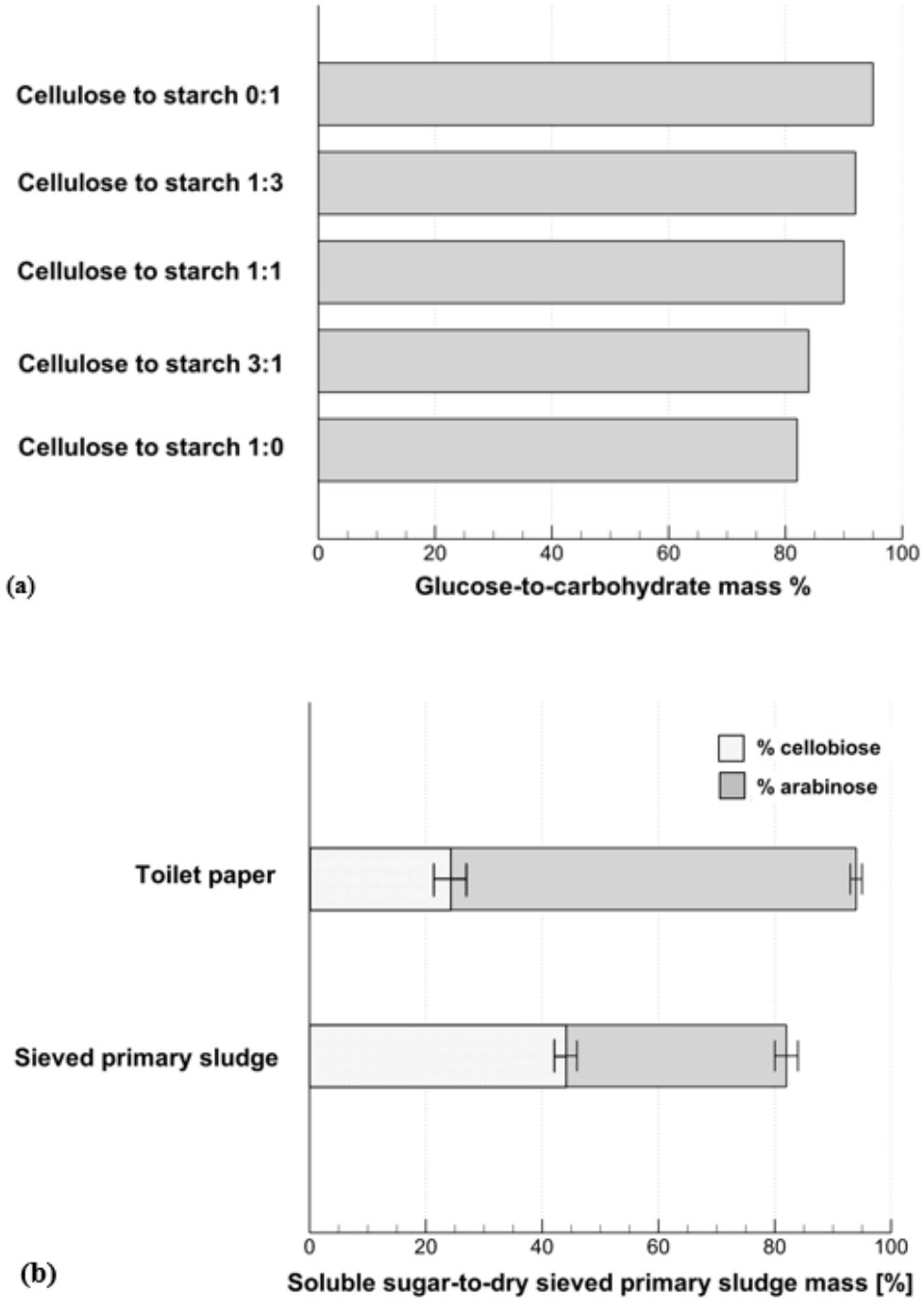

Figure 3-5. NREL method results on (a) different cellulose-to-starch ratios; (b) toilet paper and sieved primary sludge

3.3.4 Schweitzer Method

Fig. 3-6 illustrates the % cellulose by dry mass in different cellulose sources as measured by the Schweitzer method. All the tests were done in duplicates and showed excellent reproducibility as evidenced by minimal range of error bars. Toilet paper and α-cellulose were used as standards and showed 100 % recovery, which was extremely encouraging. To confirm that the reagent does not bind to starch, two additional tests were run: starch-only and, combination of starch and cellulose (50%-50% by mass). The starch-only condition recovered <1% cellulose which was anticipated, while the 50/50 starch and cellulose combination yielded 48±1 % of cellulose, re-affirming the cellulose specificity of the test method.

After the successful results obtained, the test was performed on primary clarifier sludge and sieved primary sludge which showed 18±0 %, and 37±1 % on dry basis, respectively. To further validate the method, known amounts (0.1 and 0.2 g) of α-cellulose were added to 0.3 g of dry primary sludge, and the recovery of the added α-cellulose was estimated by the difference between measured cellulose in the amended sample and raw primary sludge sample (Eq. 3.3). According to Fig. 3-6, % cellulose in standard addition test where 0.3 g of primary sludge was incorporated with 0.2 g of α-cellulose was measured to be 49±1 %. Therefore, the difference between the amended sample and the un-amended sample should be the known amount (in this case 0.2 g cellulose) should be recovered:

$$Recovered\ \%\ cellulose = \frac{Amended\ sample - primary\ sludge}{Known\ cellulose\ added} \times 100\% \qquad (3.3)$$

$$Recovered\ \%\ cellulose = \frac{(0.49 \times 0.5\ g\ amended\ sample) - (0.18 \times 0.3\ g\ primary\ sludge)}{0.2\ g\ cellulose} \times 100\% = 95\%$$

Similarly, the % cellulose in standard addition test where 0.3 g of primary sludge was mixed with 0.1 g of α-cellulose was measured to be 37±1%, i.e., 92% of added cellulose was recovered.

The Schweitzer method thus satisfies the criteria for a reliable analytical method to quantify cellulose in wastewater and sludge samples as proven based on reproducibility, accuracy and fixed 100% recovery. It is noteworthy that all other methods tested in this study with the exception of the Schweitzer method rely on measurement of soluble sugars after hydrolysis, and implicitly assume that the original concentration of soluble sugars in the samples is negligible. Furthermore, soluble sugars could be produced by hydrolysis of other carbohydrates not specifically cellulose. Thus, all other methods theoretically should overestimate the cellulose content. Despite the aforementioned, it is evident that the recoveries of cellulose using the Schweitzer method are much greater which is essentially because the Schweitzer method does not depend on the hydrolysis efficiency and reduced products analysis, but instead uses a dissolution-extraction method with gravimetric quantification of the precipitate formed. The complete recovery of both standards used i.e., toilet paper and α-cellulose, as well as the relative quickness and ease of the Schweitzer method renders it the most ideal method for cellulose determination in wastewater and sludge samples. Although, Hurwitz et al. [1961] originally developed this method for cellulose determination in sewage sludge and reported similar recovery of cellulose with high reproducibility (97.5 % and 98 %), they did not provide any proof of validation for the method. In this study, extensive validation tests using different cellulose standards such as α-cellulose and toilet paper were undertaken. Additionally, this study also confirmed that starch (another common carbohydrate found in wastewater) does not interfere with the cellulose measurements.

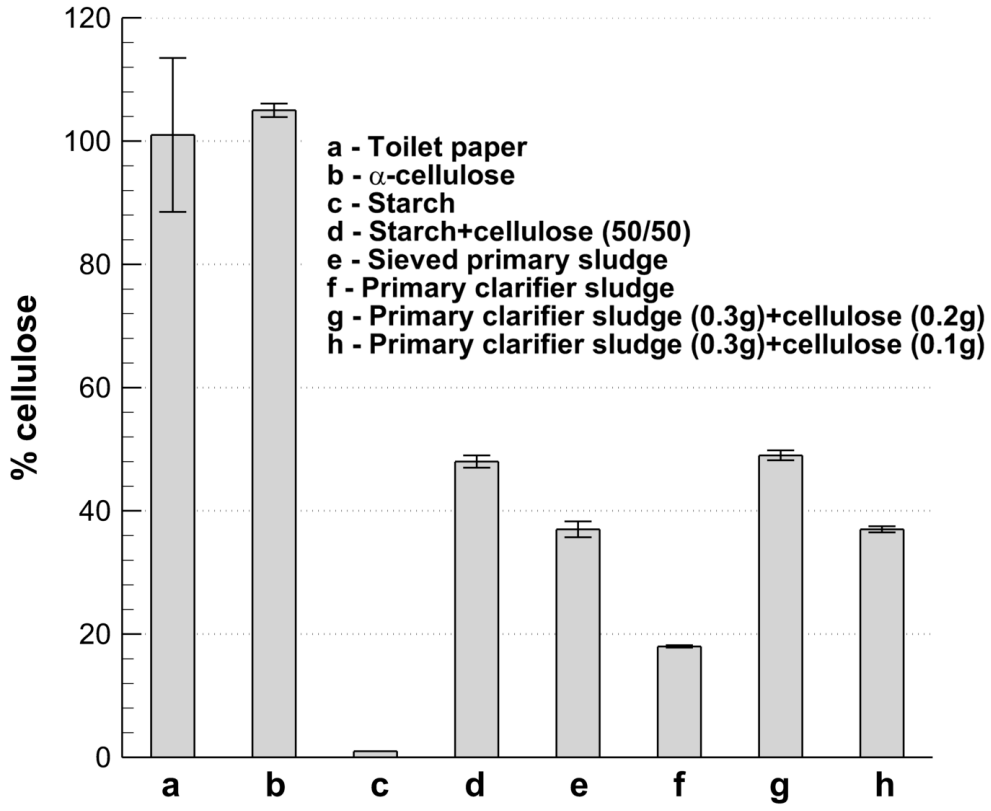

Figure 3-6. Schweitzer method results for different cellulose sources

3.4 Conclusions

After evaluation of the results obtained, it can be concluded:

- Of the four methods tested for cellulose determination in wastewater/sludges, the Schweitzer reagent method is the only reliable method.
- The advantage of the Schweitzer method is its simplicity thanks to its specificity to cellulose, reproducibility, the 100% recovery and, relative quickness of the test as well as its independence from hydrolysis reactions.
- Having a reliable method to quantify cellulose in wastewater will have great implications on wastewater research and will aid the already emerging trend to increase sustainability and resource recovery.

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Chapter 4

4 Evaluation of COD Fractionation and Biokinetic Parameters of Microsieved Wastewater

4.1 Introduction

The implementation of COD fractions and kinetic coefficients improves the effectiveness of a model to describe and predict the fate of the COD fractions throughout activated sludge processes [Tas et al., 2009]. The Activated Sludge Model (ASM) is the most widely used model for design, operation, control, troubleshooting, upgrading, modelling, and optimization of biological wastewater treatment processes [Spanjers and Vanrolleghem, 1995; Gernaey et al., 2001; Gori et al., 2011].

Respirometry is one of the oldest yet advanced experimental tools that has been used to determine COD fractionation and kinetic parameters. In a respirometry test, measurements of the oxygen uptake rate (OUR) are used to delineate these characteristics since the oxygen consumption is directly associated with COD removal and the biomass generated [Vanrolleghem, 2002]. OUR profiles are generated from an aerated batch reactor fed with a pre-determined substrate-to-biomass ratio (S_O/X_O). Typically, a series of batch tests are performed on different fractions of respective wastewater and activated sludge: (i) unfiltered wastewater, (ii) filtered (0.45 µm) wastewater, (iii) mixed-liquor alone [Xu et al., 2006; Tran et al., 2015]. Among the biokinetic parameters, biomass yield coefficient (Y_H), maximum specific growth rate (μ_{max}), decay coefficient (b_H), and substrate half-saturation coefficient (K_S), associated with ordinary heterotrophic organisms (OHOs), have been identified to be the most *influential* parameters for model calibration [Liwarska-Bizukojc and Biernacki, 2010]. The Y_H has been reported in the literature to range between 0.58-0.67 mg cell COD/mg COD removed, whereas μ_{max} ranges from 1 to 6 d^{-1} [Orhon et al., 1995; Henze et al., 2000]. The b_H is reported to range between 0.2-0.6 d^{-1} [Henze et al., 2000].

Municipal wastewaters can be fractionated into biodegradable and non-biodegradable components, where each of these fractions occur in soluble and particulate forms. Readily

biodegradable COD (S_S), rapidly hydrolysable COD (S_H), and soluble inert COD (S_I) are associated with the soluble fraction, while the slowly biodegradable COD (X_S), heterotrophic biomass (X_H), and particulate inert COD (X_I) are associated with the particulate fraction. Typically, the S_S fraction in municipal wastewater can range from 10% to 45% of the TCOD [Orhon et al., 1994; Ubay-Cokgor et al., 1998]. S_I ranges from 2% to 7% of the TCOD, and the remaining soluble fraction is the S_H [Ubay-Cokgor et al., 1998; Tas et al., 2009]. Within the particulate fraction, X_S constitutes the majority ranging from 23% to 62% of the TCOD, whereas the X_H accounts for 8%-20% of the TCOD [Ubay-Cokgor et al., 1998; Yu et al., 2010]. The X_I ranges from 7% to 29% of the TCOD [Orhon et al., 1994; Tas et al., 2009].

Medium and high-strength wastewaters usually undergo primary treatment that affects the various COD fractions with different biodegradation characteristics, which eventually affects the performance of biological processes downstream [Gori et al., 2011]. The rotating belt filter (RBF), has emerged as a viable primary treatment alternative to primary clarification (PC). The RBF removes suspended solids by microsieving and the performance of the RBF depends on the particle size distribution in the influent wastewater as well as the mesh pore size, and flow rate [Lema and Martinez, 2017]. Furthermore, the RBF technology is reported to enhance cellulose (originating from toilet-paper use) removal from wastewater [Ruiken et al., 2013]. While the COD fractionation of primary clarification effluents is widely reported in the literature [Henze et al., 2000], the fractionation of RBF effluent COD has not been reported with only few sparse studies that examined its denitrification kinetics [Razafimanantsoa et al., 2014a; Razafimanantsoa et al., 2014b]. Therefore, it is imperative to characterize the RBF effluent beyond the conventional macroscopic parameters (including total and volatile suspended solids, or biochemical and chemical oxygen demand) in order to understand the implications of integrating RBF as well as predict overall performance. The concentration of organic carbon and its biodegradability in the influent to the biological process significantly impacts the overall nutrient removal efficiency, especially for biological phosphorous removal and nitrogen removal by pre-denitrification [Tas et al., 2009; Rusten et al., 2017].

In this context, the main objective of this study was to investigate the impact of two primary treatment technologies in terms of conventional parameters as well as the assessment of the fractionation of different COD components and the biokinetic parameters that are used for model simulations, to better understand the implication of using RBF for primary treatment. Three wastewaters, that is, raw wastewater, primary clarifier effluent, and RBF effluent, were characterized using respirometric techniques.

4.2 Materials and Methods

4.2.1 Sample Collection

Raw wastewater (RWW) (screened and degritted), primary clarifier effluent (PCE) (retention time of 2 h at average flow) and return activated sludge (RAS; used as inoculum) were collected from the Greenway Wastewater Treatment Plant in London, ON (Canada). RBF effluent (RBFE) was collected from a full-scale RBF pilot (Salsnes Filter 2000 equipped with 350 µm microsieve) operated at a high hydraulic loading rate to avert cake formation. The 350 µm microsieve simultaneously optimizes filter capacity and solids retention. This pore size also corresponds to the most widely used microsieve in full-scale applications [Rusten et al., 2017]. The wastewater samples were stored at 4 ^{o}C until use within 10 days of collection. The unfiltered wastewater samples were used the same day of collection. Filtered wastewater was filtered the same day of collection prior to storing at 4 ^{o}C.

4.2.2 Respirometry Set-Up

Oxygen uptake (OU) was measured using an 8-cell Challenge Respirometer (Respirometer Systems and Application, Fayetteville, Arkansas, USA) equipped with 0.5 L batch bottles completely mixed with magnetic stirrers. The OUR measurements were used to determine the biomass yield coefficient (Y_H), readily biodegradable COD (S_S), maximum growth rate (μ_{max}), heterotrophic biomass (X_H), and endogenous decay (b_H), using the methods described by Xu et al. [2006]. The tests were set an at initial substrate-to-biomass ratio of 4 mg COD/mg VSS [Xu et al., 2006] and allylthiourea (ATU) was

added (20 mg/L) to the test bottles to inhibit nitrification. The assessment of the S_I was determined using the method developed by Orhon et al. [1994] using sequential batch reactors (SBR) fed with glucose, filtered, and unfiltered wastewater; with the same initial COD as the filtered wastewater reactor. The SBRs (1 L working volume) were operated at a SRT of infinity, fill ratio of 0.5, and one cycle per day (5 min feeding, 22.75 h react, 1 h settling followed by 10 min decanting). The remaining soluble fraction (readily hydrolysable COD, S_H), as well as the remaining particulate fractions (slowly biodegradable COD, X_S; particulate inert COD, X_I) were calculated based on the COD mass balance. The respirometer and the SBRs were conducted at room temperature (20-22 ^oC). Respirometry tests were conducted on the filtered and unfiltered wastewater samples of RWW, PCE, and RBFE. The respirometry test was run for a duration of 3-5 days until the OU plateaued, and the SBRs were operated until a stable COD was reached in the decanted effluent. Three respirometric runs (Sep 2017, Dec 2017, and Jan 2018) were conducted to validate the results as well as report the range in parameters since wastewater characteristics vary from day-to-day.

4.2.3 Nitrate Uptake Rate (NUR) Tests

Three batch NUR tests (May, July, Aug 2015) were conducted to compare the denitrification potentials of primary influent, RBF effluent, and primary clarifier effluent as the carbon source. The batch reactors (1 L) were fed with nitrates, RAS (from Greenway WWTP; used as inoculum), and respective wastewater, resulting in different TCOD/NO_3-N ratios. The tests were performed at room temperature, and the nitrates depletion and soluble COD consumption was measured over time.

4.2.4 Analytical Methods

The collected wastewater samples were analyzed for total suspended solids (TSS), volatile suspended solids (VSS) following Standard Methods [APHA, 1998]. A 0.45 µm membrane filter was used to differentiate between soluble and particulate fractions. Accordingly, total chemical oxygen demand (TCOD) and soluble chemical oxygen demand (SCOD) were measured using HACH test kits (HACH, London, Ontario, Canada). Nitrates (NO_3-N) were measured using HACH test kits.

4.3 Results and Discussion

4.3.1 Conventional Characterization

Results of conventional/routine characterization of the three wastewaters sampled at three different times are presented in Table 4-1. TSS measurements indicates that the TSS removal efficiencies of the full-scale PC and the full-scale pilot RBF were 69±3% and 28±1%, respectively, and accordingly the TCOD removal efficiency was 42±11% and 17±2%, respectively. Measurements of the TCOD and SCOD indicated that 61±5%, 40±8%, and 54±3% of the TCOD was particulate (XCOD) in nature for the RWW, PCE, and RBFE, respectively. As expected, the SCOD in the three wastewaters was similar at 306±72 mg/L, and thus, neither primary treatment impacted the SCOD fraction. The SCOD fraction in the three wastewaters followed a similar trend as that of the TSS removal efficiency, where the fraction of SCOD/TCOD fraction was the lowest for RWW (39%), and highest for PCE (60%). The observed VSS/TSS ratios of 0.78±0.03, 0.79±0.10, and 0.74±0.05 for RWW, PCE, and RBFE, respectively, were consistent with the typical ratio (0.6 to 0.8) observed in municipal wastewater [Tchobanoglous et al., 2003]. The XCOD/VSS ratio were observed to be 1.82±0.40, 2.17±0.53, and 1.95±0.44 for RWW, PCE, and RBFE, respectively, which are slightly higher than the typical ratio of 1.50 [Henze et al., 2008]. The differences observed in the VSS/TSS and XCOD/VSS ratios in the three wastewaters were statistically insignificant ($p>0.05$).

Table 4-1. Conventional characteristics of the three wastewaters at three different sampling days

Parameter	Unit	RWW					PCE					RBFE				
		#1	#2	#3	Avg	StDev	#1	#2	#3	Avg	StDev	#1	#2	#3	Avg	StDev
TCOD (C_T)	mg/L	715	826	871	804	80	389	411	621	474	128	586	674	744	668	79
SCOD (S_T)	mg/L	271	289	389	316	63	235	212	425	290	117	269	296	368	311	51
XCOD (X_T)	mg/L	444	537	482	488	46	154	200	196	183	25	318	378	377	357	34
TSS	mg/L	266	381	419	356	80	88	104	140	111	27	193	270	309	258	59
VSS	mg/L	198	302	333	278	71	61	82	125	89	33	131	204	241	192	56
ISS	mg/L	69	80	86	78	9	27	22	15	21	6	62	67	69	66	3
TSS Removal	%	na	na	na	na	na	67	73	67	69%	3%	28	29	26	28%	1%
TCOD Removal	%	na	na	na	na	na	46	50	29	42%	11%	18	18	15	17%	2%
SCOD/TCOD		0.38	0.35	0.45	0.39	0.05	0.60	0.51	0.68	0.60	0.08	0.46	0.44	0.49	0.46	0.03
VSS/TSS		0.74	0.79	0.79	0.78	0.03	0.69	0.79	0.89	0.79	0.10	0.68	0.75	0.78	0.74	0.05
XCOD/VSS		2.25	1.78	1.45	1.82	0.40	2.52	2.43	1.57	2.17	0.53	2.42	1.85	1.56	1.95	0.44

na: not applicable

#1, #2, #3 refers to the three different sampling days

Avg: average; StDev: standard deviation

Table 4-2. Summary of biokinetic parameters and COD fractionation of the three wastewaters

Parameter		Unit	RWW	PCE	RBFE
Biomass yield coefficient	Y_H	mg COD/mg COD	0.65±0.05	0.68±0.08	0.64±0.04
Decay coefficient	b_H	d^{-1}		0.40±0.04	
Maximum specific growth rate	μ_{max}	d^{-1}	2.30±0.80	3.46±0.31	2.48±0.67
Readily biodegradable COD	S_S	mg COD/L	239±29	235±93	222±18
		% of TCOD	30%±4%	46%±3%	34%±5%
		% of SCOD	76%±8%	72%±1%	73%±12%
Soluble inert COD	S_I	mg COD/L	13±3	14±2	14±3
		% of TCOD	2%±0.4%	3%±0.8%	2%±0.5%
		% of SCOD	4%±1%	5%±2%	5%±1%
Readily hydrolysable COD	S_H	mg COD/L	64±41	84±28	74±51
		% of TCOD	8%±4%	18%±4%	11%±6%
		% of SCOD	19%±9%	30%±10%	23%±13%
Heterotrophic biomass	X_H	mg COD/L	18±6	8±6	17±4
		% of TCOD	2%±0.6%	2%±0.7%	3%±0.4%
Slowly biodegradable COD	X_S	mg COD/L	250±20	93±13	180±16
		% of TCOD	31%±3%	20%±5%	27%±1%
		% of XCOD	51%	51%	50%
Particulate inert COD	X_I	mg COD/L	219±21	82±11	161±15
		% of TCOD	27%±2%	18%±4%	24%±1%
		% of XCOD	45%	45%	45%
Total inert COD	$S_I + X_I$	mg COD/L	233±23	96±14	175±17
		% of TCOD	29%±3%	21%±5%	26%±2%

4.3.2 COD Fractionation

The detailed COD fractions of the three wastewaters are depicted in Table 4-2. Y_H was calculated to be 0.66±0.02 mg COD/mg COD by plotting net oxygen consumption simultaneously with SCOD consumption in the filtered wastewater respirometer bottles (Eq. 4.1). The Y_H determined agreed with the literature which reports a range of 0.63-0.67 mg COD/mg COD [Henze et al., 2000]. Readily biodegradable COD (S_S) was experimentally determined from the OUR profile of the filtered wastewater samples. During the consumption of S_S, the OUR remains approximately constant, however, the OUR drops to a lower level when the S_S is completely depleted. The oxygen consumed before this drop is used to estimate the S_S (Eq. 4.2), for instance, the OUR profiles from Run #1 are plotted in Fig. 4-1 (Runs #2 and #3 are in Appendix B), where the S_S was depleted at ~47 h.

$$Y_H = 1 - \frac{\Delta O_2}{\Delta SCOD} \tag{4.1}$$

$$S_S = \frac{\Delta O_2}{1 - Y_H} \tag{4.2}$$

Accordingly, the S_S was determined to be 239, 235, and 222 mg COD/L (average of 232±9 mg COD/L), accounting for 30%, 46%, and 34% of the TCOD for RWW, PCE, and RBFE, respectively. Moreover, since the SCOD in the three wastewaters was similar 306±72 mg/L, 74±3% of the SCOD was S_S. Soluble inert COD (S_I) was determined from the SBRs. The SCOD profiles of RWW (filtered and unfiltered) and glucose-fed SBR from Run #1 are depicted in Fig. 4-2. The S_I fraction is the difference in the residual SCOD of the filtered wastewater SBR and glucose SBR, and accordingly the S_I was determined to be 14±0.5 mg/L for the three wastewaters corresponding to 5% of the SCOD and 2% to 3% of the TCOD. The remaining soluble fraction, S_H, was calculated by using the mass balance on the soluble fractions (Eq. 4.3) as 64, 84, and 74 mg/L, accounting for 19%, 30%, and 23% of the SCOD for RWW, PCE, and RBFE, respectively. The literature reports S_H to range anywhere from 13% to 39% of the TCOD [Orhon et al., 1999; Tas et al., 2009].

$$S_H = S_T - S_S - S_I \quad (4.3)$$

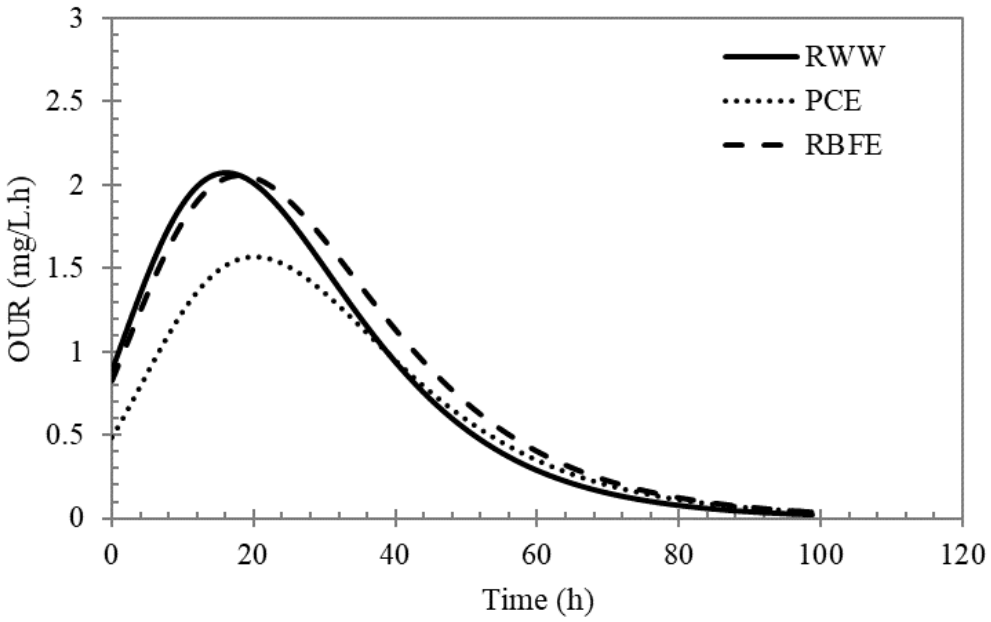

Figure 4-1. Oxygen uptake rate profile for the filtered wastewaters for Run #1

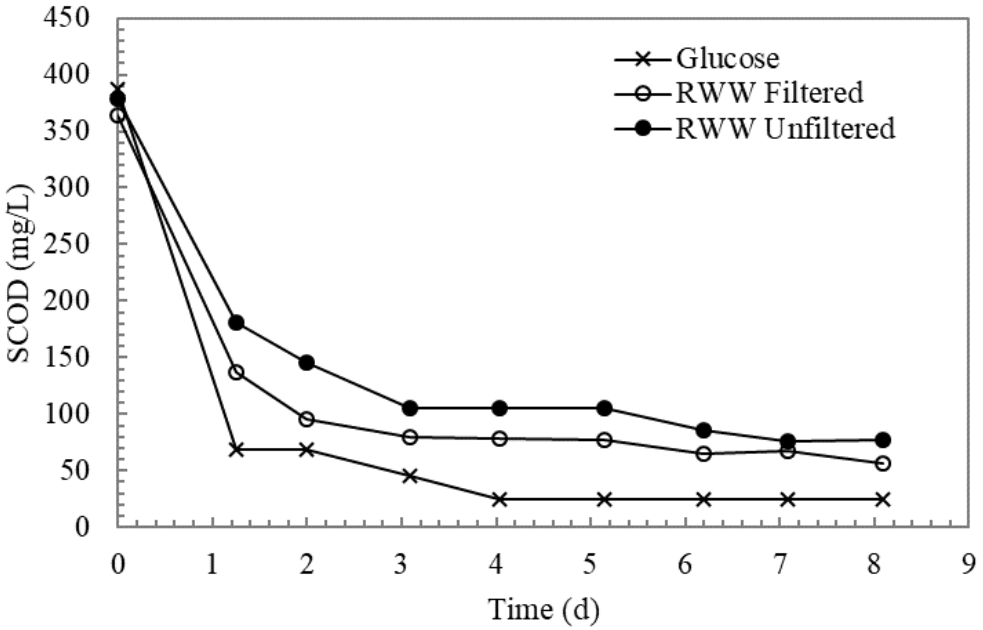

Figure 4-2. SCOD profiles for the RWW SBRs from Run #1

The decay coefficient was calculated by plotting OUR with time of the respirometer test with sludge-only and devoid of substrate (Eq. 4.4).

$$\ln OUR = -b_H t \tag{4.4}$$

The decay coefficient (b_H) of the activated sludge from the three runs was determined to be 0.40±0.04 d^{-1} which was in accordance with Henze et al. [2000]. The maximum growth rate of heterotrophs (μ_{max}) was experimentally determined from the OUR data of the filtered wastewater samples according to Eq. 4.5:

$$\ln \frac{OUR}{OUR_{initial}} = (\mu_{max} - b_H)\, t \tag{4.5}$$

The average μ_{max} were calculated to be 2.30, 3.46, and 2.48 d^{-1} for RWW, PCE, and RBFE, respectively, consistent with the 2-6 d^{-1} reported by Henze et al. [2000]. Although the S_S, responsible for growth kinetics, was similar in all three wastewaters, only the PC appears to have improved the μ_{max}.

As expected, the heterotrophic biomass (X_H) was calculated (Eq. 4.6) to be the high for RWW (18 mg COD/L) and RBFE (17 mg/L), and low PCE (8 mg COD/L), corresponding to the 2% to 3 % of the TCOD.

$$OUR_{initial} = \frac{1-Y_H}{Y_H}\mu_{max} X_H + (1 - f_e)\, b_H X_H \tag{4.6}$$

where f_e is the inert COD produced from biomass decay and a value of 0.2 g COD/g COD was used [Tchobanoglous et al., 2003].

The slowly biodegradable COD (X_S) was determined by first calculating particulate BOD_5 ($XBOD_5$) which can be obtained from the OU data of the unfiltered wastewater and filtered wastewater (Eq. 4.7). Typically, the biodegradable COD to BOD_5 ratio is 1.6 [Tchobanoglous et al., 2003], therefore, based on the BOD_5 data, biodegradable XCOD was estimated and plotted against the VSS for all the three runs of the three wastewaters as shown in Fig. 4-3.

$$XBOD_5 = BOD_5 - SBOD_5 \tag{4.7}$$

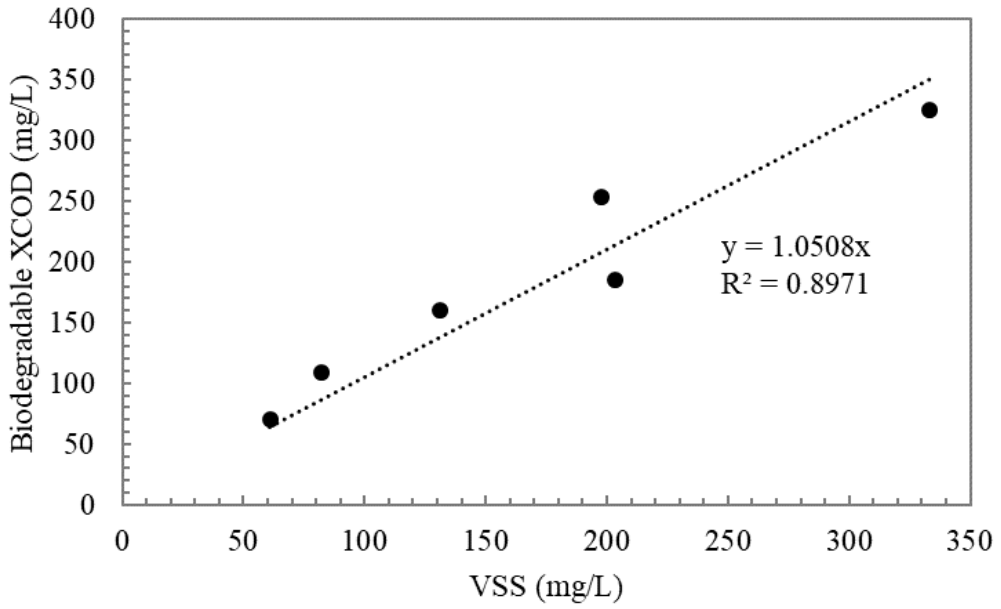

Figure 4-3. Relationship between particulate biodegradable COD and VSS

Using the above relationship and the average XCOD/VSS ratio for all wastewaters (Table 4-1, that is, 1.98), biodegradable XCOD was estimated to be 55% of the particulate COD. Accordingly, the X_S was calculated as per Eq. 4.8 and was determined to be 250±20, 93±13, and 180±16, accounting for 31%, 20%, and 27% of the TCOD for RWW, PCE, and RBFE, respectively.

$$X_S = (0.55 \times X_T) - X_H \quad (4.8)$$

The remaining X_I, was calculated by using the mass balance on the particulate fractions (Eq. 4.9) as 27%, 18%, and 24% of the SCOD for RWW, PCE, and RBFE, respectively. It is obvious that the RWW would have the most inerts whereas the PCE would have the least as majority of them settle during sedimentation.

$$X_I = X_T - X_S - X_H \quad (4.9)$$

Although, typically biodegradable XCOD is ~80% of the XCOD, the ranges observed in this study are in line with the ones reported in the literature. The literature has reported wide ranges for X_S (23%-62%) and X_I (7%-29%) fractions [Orhon et al., 1994; Orhon et

al., 1999; Ubay-Cokgor et al., 1998]. The wide range reported in the literature is because the composition of wastewater varies from day-to-day and from site-to-site. The need to collect site-specific data for COD fractionation and biokinetic parameters for better implementation of models has been emphasized by Gori et al. [2011].

4.3.3 Denitrification Potential

Three NUR tests were conducted to test the denitrification potential as well as quality of carbon source in the three wastewaters. The reduction in COD associated with nitrate utilization was used to estimate the biodegradable COD content of wastewater [Ubay-Cokgor et al., 1998; Tas et al., 2009]. Fig. 4-4 depicts the SCOD and nitrate uptake profile for the three wastewaters for Run #1 (Runs #2 and #3 are in Appendix B). Upon evaluation of the SCOD consumption during initial (fast) nitrate uptake, the S_S was estimated to be 18%±0.07%, 27%±5%, and 20%±0.01% of the TCOD for RWW, PCE, and RBFE, respectively (and 51%±3% of the SCOD). These estimates are lower than the ones reported in Table 4-2, however, the trend in terms of percent of TCOD is similar, and the differences can be attributed to different sampling times. NUR tests were performed in the Summer of 2015, whereas the respirometric tests were conducted in Fall 2017-Winter 2018. It is evident from the profile that nitrate uptake was identical in the beginning of the test and then starts to slightly deviate. Denitrification kinetics were modeled using a two-substrate model in accordance with Eq. 4.10.

$$C_t = \beta_1 e^{-k_1 t} + \beta_2 e^{-k_2 t} \tag{4.10}$$

where C_t is concentration of NO_3-N at time t; k_1 and k_2 are the initial (fast) and slow rate, respectively. The initial (fast) specific denitrification rates were comparable for all three wastewaters in the three runs, although the magnitude was different in each test (Table 4-3). The rates obtained in this study agreed with the literature studies. Naidoo et al. [1998] tested COD/N ratio of 2-9 and reported denitrification rates of 79-124 mg NO_3-N/g VSS.d using raw wastewater as the carbon source. Razafimanantsoa et al. [2014a] tested RWW and RBFE (150 µm microsieve) and obtained rates of 80 and 100 mg NO_3-N/mg VSS.d, respectively. In conclusions, the NUR tests further confirm that the S_S fraction remains unchanged irrespective of the primary treatment used. The two-substrate model

was used instead of the first-order to identify the readily biodegradable substrate, however, the initial rates from both the models were comparable (Table 4-3).

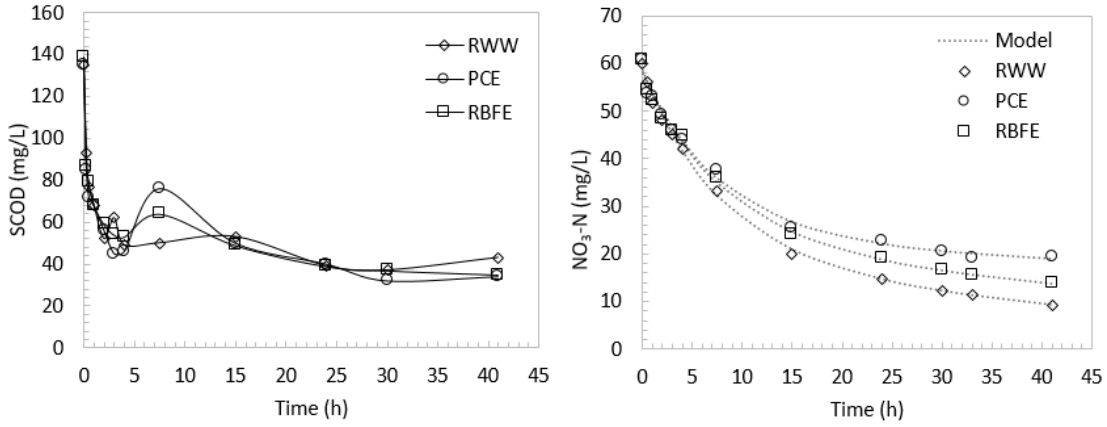

Figure 4-4. SCOD and nitrate uptake profile for the three wastewaters for Run #1

Table 4-3. Summary of the NUR tests

Run	Parameter	RWW	PCE	RBFE
Run #1	TCOD/NO_3-N	6	4	5
	SCOD/NO_3-N	2	2	2
	First-order initial rate (mg NO_3-N/g VSS/d)	72	65	67
	Two-substrate initial rate (mg NO_3-N/g VSS/d)	73	65	63
	Two-substrate slow rate (mg NO_3-N/g VSS/d)	7	2	5
	S_S (% of TCOD)	18%	31%	21%
	S_S (% of SCOD)	51%	53%	50%
Run #2	TCOD/NO_3-N	3	3	3
	SCOD/NO_3-N	1	2	1
	First-order initial rate (mg NO_3-N/g VSS/d)	189	194	193
	Two-substrate initial rate (mg NO_3-N/g VSS/d)	166	216	229
	Two-substrate slow rate (mg NO_3-N/g VSS/d)	57	57	85
	S_S (% of TCOD)	19%	28%	21%
	S_S (% of SCOD)	57%	56%	58%
Run #3	TCOD/NO_3-N	9	5	8
	SCOD/NO_3-N	3	3	3
	First-order initial rate (mg NO_3-N/g VSS/d)	224	182	228
	Two-substrate initial rate (mg NO_3-N/g VSS/d)	221	230	194
	Two-substrate slow rate (mg NO_3-N/g VSS/d)	25	86	88
	S_S (% of TCOD)	17%	21%	21%
	S_S (% of SCOD)	45%	34%	51%
	Average S_S (% of TCOD)	18±0.07%	27±5%	21±0.01%

4.4 Conclusions

Based on the experimental results obtained in this study, the following conclusions can be drawn:

- The RWW is predominantly biodegradable where 71% of the TCOD was observed to be biodegradable. PC and RBF treatment increased the biodegradable fraction to 80% and 74%, respectively, by removing inert particulates by settling and sieving, respectively.
- As expected, microsieving and settling do not impact the soluble components in the wastewaters as reflected by the same S_S, S_I, and S_H, for RWW, PCE, and

RBFE. The S_S accounted for 30%, 46%, and 34% of the TCOD for RWW, PCE, and RBFE, respectively.

- The fractionation of the particulate COD was comparable between the three wastewaters, where 55% of the particulate COD was biodegradable.
- The X_S accounted for 31%, 20%, and 27% of the TCOD for RWW, PCE, and RBFE, respectively.
- The NUR tests confirmed that the readily biodegradable COD fraction remains unchanged irrespective of the primary treatment.

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Chapter 5

5 Microsieving Raw Wastewater for Nitrogen Removal and Control in Wastewater Resource Recovery Facilities

5.1 Introduction

Nutrient discharges from wastewater resource recovery facilities (WRRFs) have been reported to severely impair aquatic life and water quality in sensitive receiving bodies by promoting eutrophication and frequent algal blooms [EPA, 2008]. When activated sludge processes are designed for biological nutrient removal (BNR), the quality and availability of the organic carbon is very important to overall operational cost, nutrient removal efficiency, and resource recovery potential [Gori et al., 2011]. There are two functionally different design philosophies for BNR plants: the first one, typically applied to higher strength or high C:N ratio wastewaters, relies on the use of a primary clarification step which is intended to remove as much particulate as possible thus allowing mostly soluble biodegradable carbon to be exploited in the denitrification stage [Tchobanoglous et al., 2003]; in the second approach, usually practiced for low-strength wastewater, the primary clarification is omitted altogether, to avoid the risk of producing a carbon-limited primary treated effluent for the downstream biological nutrient removal processes [Ubay-Cokgor et al., 2005]. However, the disadvantage of such a design option is the increased solids loading on the secondary clarifiers, and increased aeration costs [Gori et al, 2013].

In a WRRF, carbon diversion to anaerobic digestion is important for energy recovery, and reduced costs of solids treatment and disposal [Jimenez et al., 2015]. While primary clarification or high-rate activated sludge processes (such as the A-stage process) achieve carbon diversion, they are relatively high capital cost, difficult to retrofit to existing processes, and have a hydraulic limit imposed by gravity sedimentation [Lessard and Beck, 1988]. Moreover, soluble carbon can be bio-absorbed by A-stage biomass thus competing for the same substrate required for denitrification. High-rate, lower efficiency physical solids removal processes capable of achieving particulate removal, in a compact footprint, are receiving increased attention as they combine the advantages of the two

aforementioned design philosophies [Oleszkiewicz, 2015], diverting a smaller fraction of particulate organics, while leaving soluble organics available in the mainline for denitrification. In particular, the use of microsieving technologies, engineered in rotating belt filters (RBFs), has emerged as a valid primary treatment alternative for BNR plants as they allow energy recovery via anaerobic digestion and minimal diversion of readily biodegradable carbon contributing to denitrification, in a very small footprint, for a straightforward retrofit option. As such, RBFs can be seen as a primary treatment option that is compatible with BNR plants fed with both lower and higher strength domestic wastewaters. Moreover, the performance of RBFs has been validated in a number of full and pilot scales installations operating in multiple geographies [Franchi and Santoro, 2015; Caliskaner et al., 2014, Rusten et al., 2017]. Additionally, unlike primary clarifiers and A-stage clarifiers, which are detrimentally impacted by hydraulic overloads, RBF microsieving enables rapid and dynamic process control. As such, it is potentially a key process for energy-saving alternative schemes [Scott et al., 2015; Gikas, 2017]. However, RBFs selectively remove different compounds, specifically fibrous solids, including toilet paper fibers [Ruiken et al. 2013]. This may have an impact on the core capability to remove nitrogen in the main treatment plant. In order to enable the successful integration of RBF into BNR schemes, it is critical to evaluate their impact on downstream biological treatment process [Rusten et al., 2016]. Razafimanantsoa et al. [2014a] observed no impact of mesh microsieves (ranging from 1.2 µm to 150 µm) on the different specific denitrification rate (SDNR). Rusten et al. [2016] conducted a similar study in moving bed biofilm reactors (MBBRs) where one reactor was fed with 2 mm screened wastewater and another through 33 µm RBF, and not only did not observe any differences in denitrification rates for both the reactors, but also 10%-15% higher nitrification rates in the 33 µm RBF effluent reactor compared to the MBBR fed with 2 mm screened wastewater.

No previous study has been identified in the literature comparing the impact of RBF, primary clarification, and no-primary, on BNR performance. In light of continually increasing interest in carbon diversion technologies while meeting water quality targets, it is essential to understand the role of RBF on the performance of WRRFs. To address these existing gaps and the definite paucity of information on the performance of the RBF

as a primary treatment stage in BNR treatment, in this paper, two primary treatment options (microsieving and primary clarification) have been evaluated against the no-primary treatment scenario in parallel sequencing batch reactors (SBR). In particular, the impact on effluent quality caused by the two primary treatment alternatives was investigated with the goal of determining how these two processes would compete for carbon substrates used for denitrification and carbon recovery potential. Finally, process simulations were conducted and validated against experimental data collected during the SBR studies. Model-based analysis was subsequently conducted on two treatment scenarios differing in terms of wastewater strength and RBF solids capture efficiency in the primary stage.

5.2 Materials and Methods

5.2.1 Sample Collection and Preparation

Raw wastewater (RWW) (screened and degritted) and primary clarifier effluent (PC) (retention time of 2 h at average flow) were collected from the Greenway Wastewater Treatment Plant (WWTP) in London, ON (Canada) twice a week for 24 weeks (July to December 2016). Laboratory simulation of the RBF effluent was achieved by microsieving a sufficient volume of wastewater through 350 µm (identical to the one used in commercially available full-scale RBF) microsieve to emulate operation at a high hydraulic loading rate to avert cake formation in the full-scale system. The 350 µm microsieve pore size is justified based on the need for simultaneously optimizing filter capacity and solids retention. This pore size also corresponds to the most widely used microsieve in full-scale applications [Rusten et al., 2017]. The wastewater samples were stored at 4 ^{o}C until use, which occurred within 72 hours. The influent characteristics of the three SBRs, RWW SBR, PC SBR, and RBF SBR, are provided in Table 5-1.

Table 5-1. Influent characteristics of the three SBRs

Parameters	Unit	RWW SBR*	PC SBR*	RBF SBR*
TSS	mg/L	330 ± 67	100 ± 14	240 ± 54
VSS	mg/L	260 ± 55	80 ± 14	180 ± 41
TCOD	mg/L	750 ± 164	490 ± 155	610 ± 138
sCOD	mg/L	310 ± 113	330 ± 145	300 ± 98
TN	mg/L	56 ± 12	46 ± 14	52 ± 11
NO_3-N	mg/L	0.66 ± 0.3	0.55 ± 0.3	0.61 ± 0.3
NO_2-N	mg/L	0.24 ± 0.3	0.02 ± 0.01	0.25 ± 0.3
NH_4^+-N	mg/L	36 ± 8	34 ± 10	34 ±7
TP	mg/L	9 ± 2	6 ± 1	9 ± 2
Alkalinity	mg $CaCO_3$/L	400 ± 65	400 ± 70	390 ± 54
pH	-	7.5 ± 0.5	6.9 ± 0.6	7.2 ± 0.4
TCOD/TN	-	13 ± 2	10± 2	11± 2
sCOD/TN	-	6 ± 3	7 ± 3	6 ± 3

*Averages and standard deviations for 22 samples of RWW, 22 of PC, and 24 of RBF

5.2.2 SBR Set-Up and Operation

Three laboratory-scale anoxic-aerobic SBRs with a working volume of 2 L were seeded with sludge from the nitrifying Greenway WWTP. The SBRs were operated with a cycle time of 6 h, that is, four cycles per day, at room temperature (22-24 ^oC). Each cycle consisted of 10 min anoxic fill, a 1.25 h anoxic react period and a 3.5 h aerobic period (DO ~ 3-4 mg/L), followed by 1 h settling and 0.25 h decanting. A fill ratio of 0.35 (i.e., V_{fill}/V_{total}) was used. Hence, residual nitrate from the 0.65 fill was removed by organics in the fresh feed, with some nitrate discharged in the decant period. While this results in elevated effluent nitrate levels, it effectively exposes kinetics of nitrification and denitrification due to the batch nature. The MLSS wasting rate was 0.2 L/d to maintain a solids retention time (SRT) of approximately 10 d.

Cyclic studies with measurements of liquid-phase components were carried out regularly during both the start-up and the steady-state operation to monitor the performance of the SBRs as well as to determine the specific nitrification and denitrification rates. Aliquots 10 mL in volume were withdrawn at predetermined time intervals and the following parameters were analyzed: total COD (TCOD); soluble COD (SCOD); NH_4-N; NO_3-N; NO_2-N; soluble phosphorus.

5.2.3 Monitoring, Sampling, and Analysis

The DO and pH in each SBR were measured with a DO probe and a pH probe, respectively. Mixed liquor samples were collected from the SBR periodically and analyzed for total suspended solids (TSS), volatile suspended solids (VSS), and total nitrogen. Influent and effluent samples were collected from the SBR periodically and were analyzed for inorganic nitrogen species (ammonia nitrogen, NH_4-N; nitrate nitrogen, NO_3-N; nitrite nitrogen, NO_2-N), total nitrogen (TN); total phosphorus, (TP); soluble chemical oxygen demand (sCOD); total chemical oxygen demand (TCOD) using portable Hach test kits (HACH, London, Ontario). Additionally, the influent and effluent samples were also analyzed for total suspended solids (TSS), volatile suspended solids (VSS) and alkalinity based on Standard Methods [APHA, 1998].

5.2.4 Model-Based Analysis

A model-based analysis of the lab-scale SBR was performed using GPS-X ver 6.4 (Hydromantis, Inc. 2014). The mantis model, which is an extension of the ASM1 model, was used for the biological process. The mantis model involves 2 modifications of ASM1: (i) growth of autotrophic and heterotrophic microorganisms to describe growth under low ammonia and high nitrate conditions, and (ii) hydrolysis of rapidly biodegradable substance [Lopez-Arenas et al., 2003, Mulas, 2006]. The TSS/COD influent model was applied for influent characterization.

The RWW wastewater collected in this study is considered medium-strength (MS) wastewater with TSS of ~300 mg/L. The calibrated model was further used to evaluate a scenario with high-strength (HS) RWW characteristics (TSS=500 mg/L) and how it would impact RBF performance, and in turn, SBR performance. Since, GPS-X allows to further fractionate the organic variables including particulate and soluble inerts (Xi and Si), slowly and readily biodegradable substrate (Xs and Ss), and organic nitrogen components; HS RWW characteristics were generated using the fractionation and coefficients obtained from the MS RWW characteristics as shown in Table 5-2a. The soluble components were maintained the same, and the particulate components were calculated using the stoichiometric ratios of VSS/TSS, VSS/XCOD, Xi/XCOD,

Xs/XCOD, etc. reported in Table 5-2a. For the RBF effluent, it is assumed, based on literature [Franchi and Santoro, 2015], that the TSS removal efficiency of RBF increased to 70%, with no removal of soluble substrates. Using the same ratios, the RBF effluent was calculated accordingly (Table 5-2b).

Table 5-2a. Organic variables and coefficients of the medium-strength RWW obtained from GPS-X 6.4

Parameter	Input
Readily biodegradable COD, Ss	249.2
Inert soluble COD, Si	36.7
Ammonia, NH_4	39.2
Soluble phosphorous, sP	8
Biodegradable organic nitrogen, bON	1.68
Inert particulate COD/particulate COD, Xi/XCOD	0.18
Slowly biodegradable COD/particulate COD, Xs/XCOD	0.82
Particulate biodegradable organic nitrogen/slowly biodegradable COD, pbON/Xs	0.0268
Refractory particulate organic nitrogen/inert particulate COD, rpON/Xi	0.068
VSS/TSS	0.78
Particulate COD/VSS, XCOD/VSS	1.71
Particulate phosphorous/particulate COD, pP/XCOD	0.00625

Table 5-2b. Characteristics of generated HS RWW

Parameter	High strength RWW	RBF effluent (assumed 70% removal)
TSS	500	150
VSS	390	117
Particulate COD, XCOD	667	200
Inert particulate COD, Xi	120	36
Slowly biodegradable COD, Xs	547	164
TCOD	953	486
Particulate biodegradable organic nitrogen, pbON	14.7	4.4
Refractory particulate organic nitrogen, rpON	8.2	2.4
Total Kjeldahl nitrogen, TkN	63.7	47.7
TP	12	9

5.3 Results and Discussion

5.3.1 Primary Treatment Performance

The TSS removal efficiency for microsieved wastewater ranged from 16% to 46% and averaged 27% while that of primary clarification ranged from 56% to 82% and averaged 67%. The substantial variation in the removal efficiencies is due to the day-to-day variability in the wastewater TSS, as can be seen in Fig. 5-1a (round black dots). Additionally, it was observed that the RBF removed up to 7% of TN and 18% of TCOD, whereas the primary clarifier removed up to 20% of TN and 32% of TCOD. The removal efficiencies achieved in this study were in agreement with the results observed by Rusten et al. [2017] where TSS removal of 25% to 48% was observed for influent TSS ranging from 160 to 400 mg/L at similar operating conditions (no cake formation). The abovementioned authors also emphasized that the removal efficiencies are dependent on influent suspended solids concentrations, specifically, higher TSS removal efficiency is observed at high influent TSS concentrations. Razafimanantsoa et al [2014b] used different microsieve openings ranging from 18 µm to 150 µm and observed TSS removal ranging from 27% (using 150 µm) to 65% (using 18 µm sieve). Furthermore, the aforementioned authors reported TN removal ranging from 5% (using 150 µm) to 15% (using 33 µm sieve) and TCOD removal ranging from 25% (using 150 µm) to 46% (using 18 µm sieve). It is important to note that the TCOD/TN ratios (Table 5-1) for the three streams were different and as expected the average TCOD/TN ratio for RWW was the highest at 13 (ranged from 9-18), compared to PC at 10 (ranged from 6-14), and RBF at 11 (ranged from 8-16). The sCOD concentration in the three wastewaters remained unchanged since neither primary treatment affected the soluble fraction and accordingly the sCOD/TN ratio of RWW, PC, RBF was observed to be 6 ± 2, 7 ± 3, and 6 ± 3, respectively.

Other high-rate primary processes, such as high-rate ballasted clarification, can achieve higher TSS removal (85% to 95%); where their performance is highly dependent on the coagulating and ballasting agents. Chemical coagulants such as alum or ferric salts, and ballasting agents such as magnetite, microsand, and recycled sludge have been reported for enhanced primary treatment processes [EPA, 2013; Lema and Martinez, 2017]. It

should also be pointed out that, in a recent study from Rusten et al. [2017] on chemically-enhanced RBF, the TSS removal efficiency increased from 40%-50% to 60%-70% with the addition of a cationic polymer in low dose upstream of the RBF, as is with the case with chemically-enhanced PC where the TSS removal efficiency can be increased up to 80%-90% with a combination of iron or aluminum salts and polymer [Lema and Martinez, 2017].

5.3.2 SBR Performance

The temporal variations of SBR influent and effluent TSS and COD are presented in Fig. 5-1a and 5-1b, respectively. Effluent characteristics of the three SBRs during steady-state operation are summarized in Table 5-3. Effluent SBR TSS concentrations from the three reactors averaged approximately 11, 16, 13 mg/L corresponding to TSS removal efficiencies of 97%, 85%, and 94% for RWW, PC, and RBF, respectively (Fig. 5-1a). Similarly, the influent and effluent TCOD characteristics are illustrated in Fig. 5-1b. The three SBRs achieved good TCOD removal efficiencies, averaging 94%, 90%, and 92% for RWW, PC, and RBF, respectively. Similar TSS (83%-97%) and TCOD (84%-91%) removal efficiencies were observed by Razafimanantsoa et al. [2014b] who operated SBRs fed with degritted wastewater sieved with meshes of 1.2, 18, 33, 50, 90, and 150 µm (SRTs ranging from 12 to 18 days). Rusten et al. [2016] achieved 91% TCOD removal efficiencies in MBBRs fed with 2 mm-sieved wastewater as well as in the reactor fed with 33 µm-sieved wastewater.

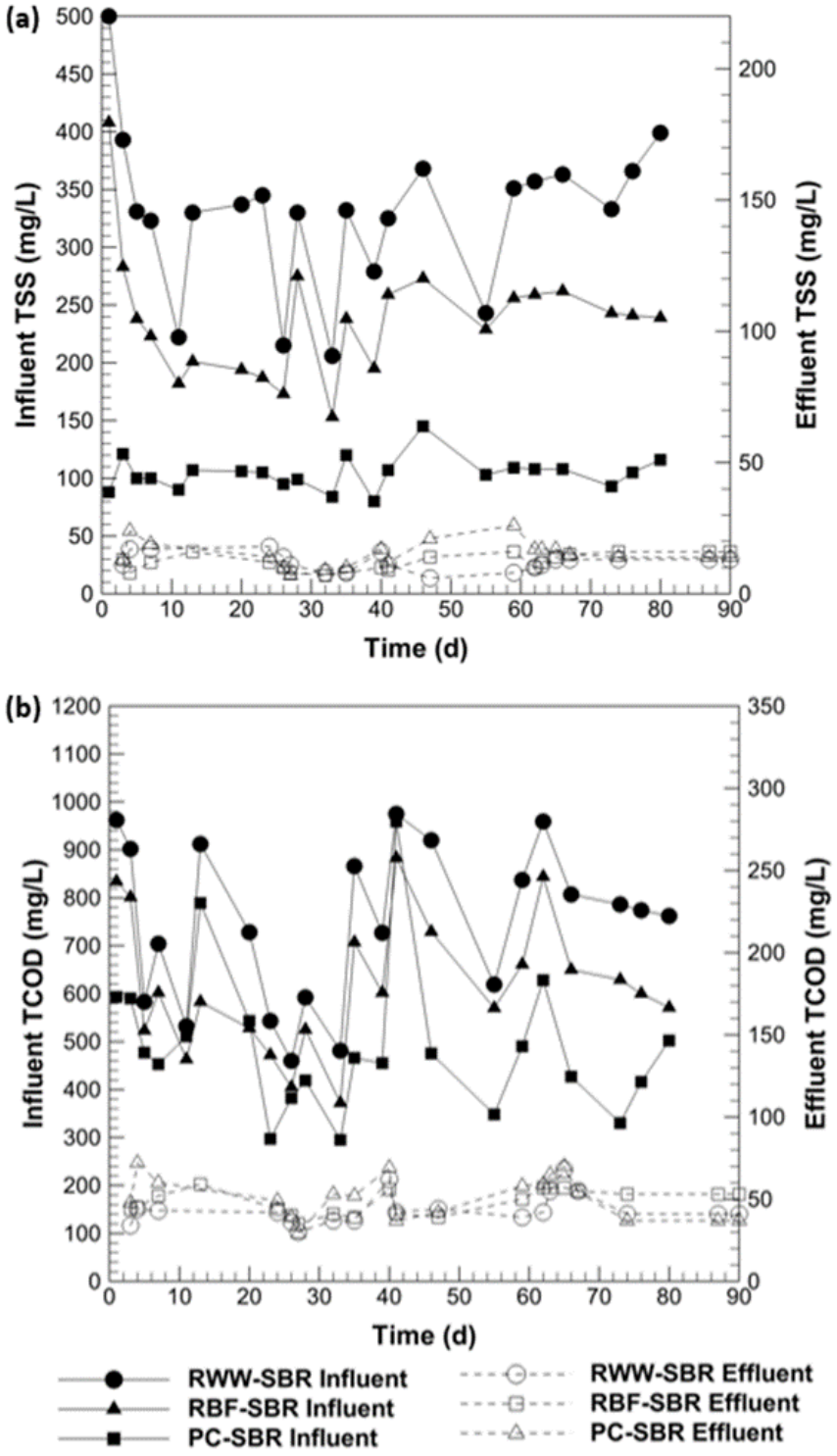

Figure 5-1. SBR Performance: Influent and Effluent (a) TSS; (b) COD concentrations

Table 5-3. Effluent characteristics of the three SBRs

Parameters	Units	RWW SBR*	PC SBR*	RBF SBR*
TSS	mg/L	11 ± 3	16 ± 4	13 ± 4
VSS	mg/L	9 ± 3	12 ± 5	9 ± 2
TCOD	mg/L	46 ± 10	51 ± 12	50 ± 7
sCOD	mg/L	39 ± 7	41 ± 10	39 ± 6
TN	mg/L	15 ± 2	15 ± 3	15 ± 2
NO_3-N	mg/L	13 ± 3	13 ± 3	13 ± 2
NO_2-N	mg/L	0.21 ± 0.1	0.29 ± 0.2	0.11 ± 0.1
NH_4^+-N	mg/L	2.11 ± 0.8	2.01 ± 0.8	1.99 ± 0.9
TP	mg/L	3 ± 2	3 ± 2	3 ± 2
Alkalinity	mg$CaCO_3$/L	280 ± 29	270 ± 37	280 ± 20
pH		7.6 ± 0.7	7.0 ± 0.4	7.1 ± 0.3

*Averages and standard deviations of 13 samples of RWW, PC, and RBF

Fig. 5-2b and 5-2c present the experimental results for the concentrations of NH_4^+-N and NO_3-N throughout the experimental period. Despite higher influent TN for RWW and RBF compared to PC (Fig. 5-2a and Table 5-1), effluent TN was the same (~15 mg/L) in all three SBRs (Table 5-3). On an average 13 mg NO_3-N/L were observed in the effluent for the three wastewaters. The residual nitrates in the effluent were an artifact of operating the SBRs in pre-anoxic mode and decanting the effluent after the aerobic phase. This is a disadvantage of SBRs, and while more complex operation can minimize effluent nitrogen, residual is elevated (compared with continuous processes). Ammonia concentration in the influent ranged from 34 to 36 mg NH_4^+-N/L whereas the concentrations in the effluent were below 2 mg NH_4^+-N/L, well below the typical site-specific limits in Ontario which vary from 3 to 10 mg/L NH_4^+-N [Eini et al., 2017], corresponding to 94% nitrification efficiency. Nitrites in the influent and effluent were negligible (<0.5 mg NO_2-N/L). Overall TN removal for the three SBRs was comparable and averaged at 73%, 68%, and 71% for RWW, PC and RBF, respectively. The TN removal efficiencies observed in this study are in line with the study reported in Razafimanantsoa et al. [2014b], who tested different sieved degritted wastewater including 33, 50, 90, and 150 µm sieves, and reported nitrogen removal efficiencies ranging from 57%-63%. The abovementioned authors observed a decline in nitrogen

removal efficiency only in SBRs fed with 1.2 µm-filtered (31%) and 18 µm-sieved degritted wastewater (40%) compared to 68% nitrogen removal with unfiltered degritted wastewater. The authors attributed this decline in performance to limited sCOD concentrations, (sCOD/TN ratio of 4.7 and 3.5 for 1.2 µm-filtered and 18 µm-sieved wastewater, respectively in the feed) and absence of hydrolysis of particulate matter (TCOD/TN ratio of 4.7 and 6.8 for 1.2 µm-filtered and 18 µm-sieved wastewater, respectively, versus 11.3 for unfiltered wastewater). Additionally, Rusten et al. [2016] observed 66% and 68% TN removal in a MBBR using 33 µm-sieved wastewater and 2 mm-screened wastewater, respectively, which is also in line with the results of this study. Based on the results of this study as well as Razafimanantsoa et al. [2014b] and Rusten et al. [2016], it can be concluded that within the range of sieve openings of 33 µm to 350 µm, there is no significant impact on nitrogen removal efficiency and the COD present in the microsieved wastewaters is sufficient for nitrogen removal. It is evident from Fig. 5-2 that after about 90 days of operation, all three SBRs demonstrated stable nitrification, denitrification, and biological nitrogen removal efficiencies despite the high variability in influent ammonia and TKN concentrations.

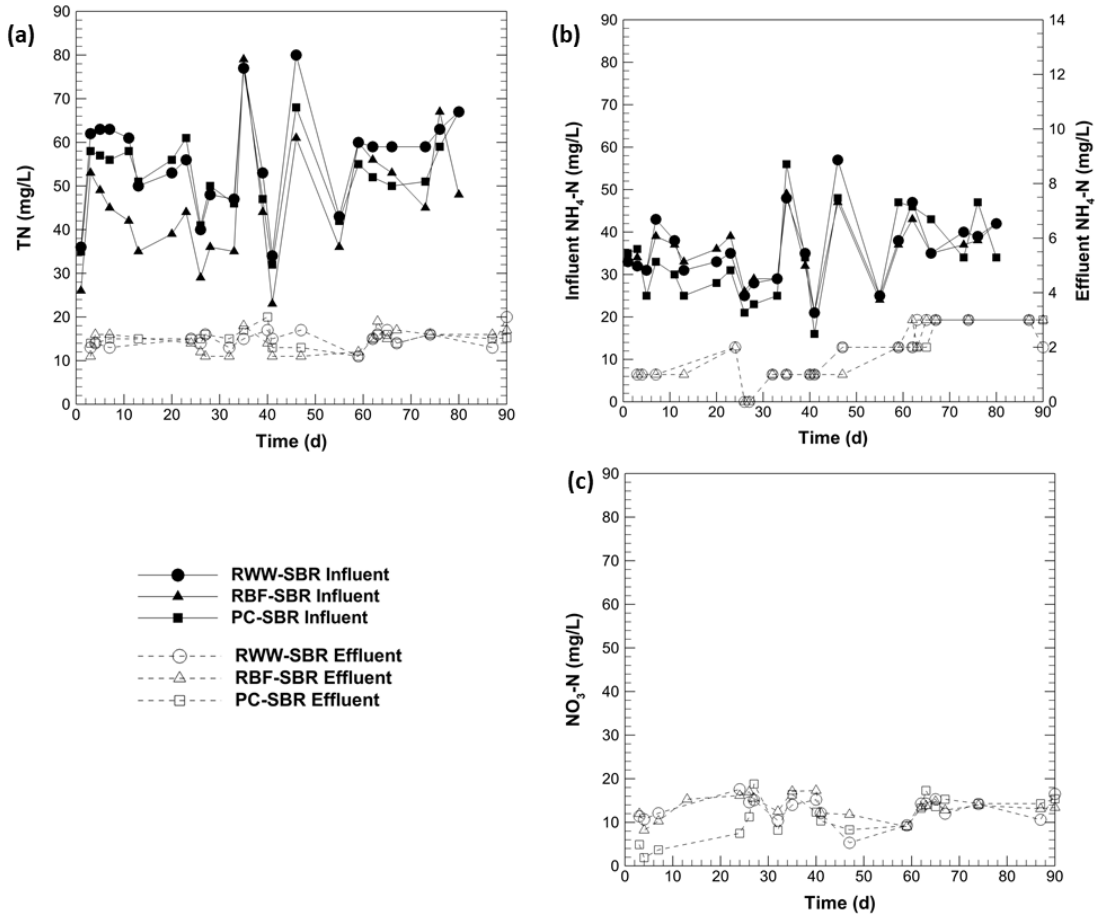

Figure 5-2. SBR performance: Influent and effluent (a) TN; (b) NH_4-N; and (c) residual NO_3-N in the effluent

5.3.3 Sludge Production and Biomass Yield

The steady-state concentrations of MLSS in the three SBRs, RWW, PC, RBF, (Table 5-4) were observed to be 3205±285, 1342±46, and 2788±237 mg VSS/L, respectively; which suggests that RBFs (operating at low TSS removal efficiency) reduce secondary clarifier solids loading by 13% compared to no-primary (i.e., RWW) case, whereas PC reduces the load by 58%. The volatile fraction of MLSS in PC SBR (0.76) was observed to be higher than the RWW (0.70) and RBF (0.73) SBR potentially due to the accumulation of inert inorganic suspended solids in the RWW and RBF SBRs. The observed biomass yields for secondary sludge derived from the linear fits of cumulative VSS production versus COD removed (not shown here; R^2 of 0.999, 0.998, and 0.994 for

RWW, PC, and RBF SBRs, respectively) were 0.23, 0.20 and 0.29 mg VSS/mg $COD_{consumed}$ for the RWW, PC, and RBF SBRs, respectively (Table 5-4). The biomass yields achieved in this study were similar to the results observed by Razafimanantsoa et al. [2014b] where biomass yield of RBF SBR (90 µm and 150 µm-sieved) was higher (0.27-0.29 mg VSS/mg COD) than for RWW SBR (0.21 mg VSS/mg COD). Sludge production was calculated based on the % TSS removal by respective primary treatment and sludge wastage rates from the SBRs. Although the biological sludge production with primary clarification system (270 mg TSS/d) was lower than the RBF system (560 mg TSS/d), the overall primary and biological sludge produced by the RBF system was 9% lower than the primary clarifier (810 mg TSS/d versus 890 mg TSS/d). This is because primary treatment removes particulate organic matter physically, as opposed to biodegradation, where only a fractional amount of the oxidized organics ends up as biomass. Thus, solids and efficient carbon diversion in primary treatment will inevitably lead to higher overall sludge production. Therefore, RBF has lower overall sludge production compared to PC despite the higher biomass yield in the RBF SBR concomitant with low TSS removal in the primary treatment.

Table 5-4. Concentrations of MLSS and MLVSS

SBR	RWW SBR	PC SBR	RBF SBR
MLSS (mg/L)	3205 ± 285	1342 ± 46	2788 ± 237
MLVSS (mg/L)	2228 ± 198	1036 ± 38	2025 ± 147
MLVSS/MLSS	0.70	0.76	0.73
Sludge production			
Primary sludge production (mg TSS/d)	na	620	250
Biological sludge production (mg TSS/d)	640	270	560
Overall sludge production (mg TSS/d)	640	890	810
Biological sludge yield (mg VSS/mg $COD_{consumed}$)	0.23	0.20	0.29

5.3.4 Steady-State Nitrogen Balance

Nitrogen balances were calculated considering the nitrogen assimilated in the biomass as well as the nitrogen denitrified based on influent TN and effluent TN (Eq. 5.1 and 5.2).

$$N\ in\ biomass = \left(N\ content\ of\ biomass, \frac{mg\ N}{mg\ MLVSS}\right)\left[Y, \frac{mg\ MLVSS}{mg\ COD_{consumed}}\left(COD_{in} - SCOD_{eff}\right)\right] \quad (5.1)$$

$$N\ denitrified = TN_{in} - N_{biomass} - TN_{effleunt} \quad (5.2)$$

In Fig. 5-3 it can be seen that the percentages of nitrogen remaining in the effluent were 27%, 32%, and 29% of the total influent nitrogen for the RWW, PC, and RBF SBRs, respectively. Similarly, nitrogen assimilated in the biomass was 27%, 19%, and 30% of the influent TN for the RWW, PC, and RBF SBRs, respectively. Correspondingly, the amount of oxidizable nitrogen in the three SBRs was comparable at 38±2 mg N/L. The percentages of nitrogen removed by denitrification were found to be 46%, 48%, and 41% for the RWW, PC, and RBF SBRs, respectively. Accordingly, the amount of COD removed anoxically accounted for 150, 157, and 130 mg COD/L for the RWW, PC, and RBF SBR, respectively.

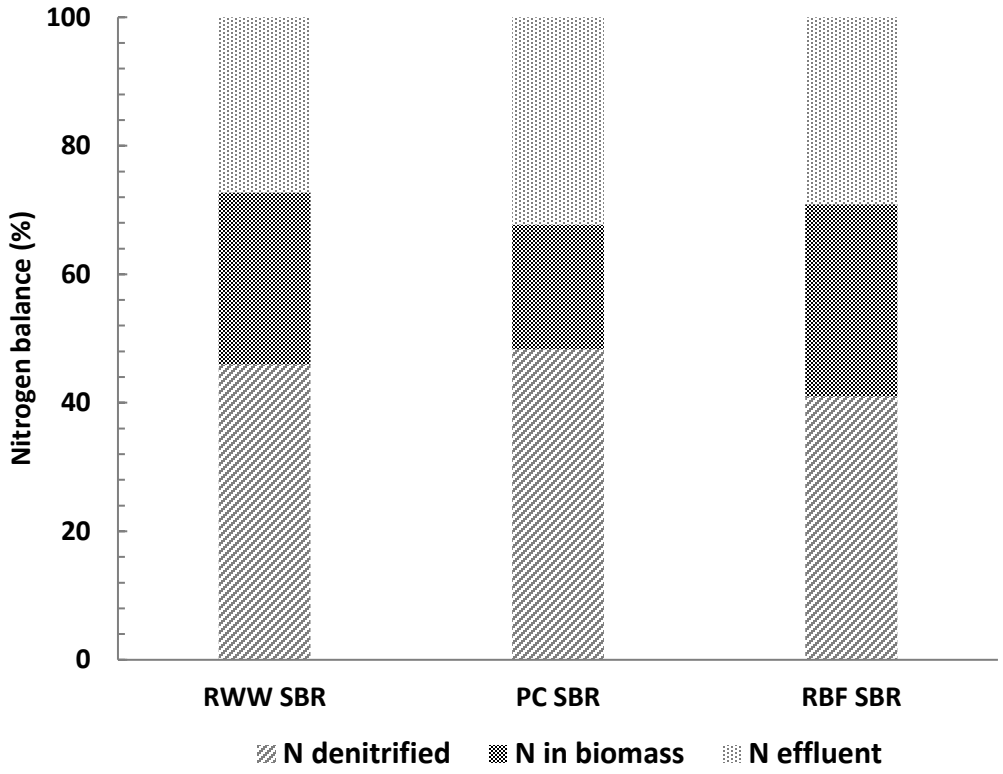

Figure 5-3. Characteristics of Nitrogen Balance

5.3.5 Nitrification and Denitrification Rates

The specific nitrification rate (SNR) and denitrification rate (SDNR) for the different SBRs are summarized in Table 5-5. The SNRs and SDNRs were calculated based on both the total MLVSS estimated using GPS-X modelling described later. The SNRs ranged from 72 to 205 mg NH_3-N/g $MLVSS_{total}$/d in the three SBRs. It can also be observed that the SNRs increased with respective primary treatment performance. For instance, PC SBR had the highest rate (205 mg NH_3-N/g $MLVSS_{total}$/d), which had the lowest SS, whereas, RWW SBR had the lowest rate. This is likely due to removal of otherwise degradable soluble organic material, which would cause heterotrophic competition with autotrophic nitrification. Razafimanantsoa et al. [2014b] observed a similar trend, that is, SNRs increased with decreasing C/N ratios. The observed SNR for raw wastewater (72 mg NH_3-N/g $MLVSS_{total}$/d) in this study was higher than what Razafimanantsoa et al. [2014b] observed (44±9 mg NH_3-N/g VSS/d).

The SDNRs observed in this study ranged from 176 to 414 mg NO_3-N/g $MLVSS_{total}$/d and 145-196 mg NO_x-N/g $MLVSS_{total}$/d. Razafimanantsoa et al. [2014b] reported rates in the range of 48-54 mg NO_x-N/g VSS/d which were lower than the rates observed in this study, despite operating at similar TCOD/TN ratios ranging between 9 and 14. The SDNRs for RWW SBR and RBF SBR were comparable at 186±14 mg NO_3-N/g $MLVSS_{total}$/d, which suggests that the removal of SS from the RWW through microsieving did not have a significant affect on the SDNR. Razafimanantsoa et al. [2014a] reported rate of 100 mg NO_3-N/g $MLVSS_{total}$/d at TCOD/TN ratio of 6 using 150 µm-sieved wastewater. Naidoo et al. [1988] reported rates in the range of 72-175 mg N/ g VSS.d using RWW and centrifuged-RWW (~43% COD removal) as the carbon source for TCOD/N ratio ranging from 2-9. Similarly, Onnis-Hayden and Gu [2008] reported SDNR ranging from 113-153 mg N/g VSS.d using glycerol-based MicroC^{TM} as the carbon source at TCOD/TN ratio of 6. It is evident from the literature, that the rates determined in this study are consistent with those reported in the literature.

Table 5-5. Specific nitrification and denitrification rates

	RWW SBR	PC SBR	RBF SBR
Specific Nitrification Rate (mg NH_3-N/g $MLVSS_{total}$/d)	72	205	89
Specific Denitrification Rate (mg NO_3-N/g $MLVSS_{total}$/d)	176	414	196
Specific Denitrification Rate (mg NO_x-N/g $MLVSS_{total}$/d)	196	145	174
Total MLVSS (mg/L)	2408	1019	2037

5.3.6 Bioprocess Modelling and Validation

The SBR performances were modelled using GPS-X (version 6.4). The model was validated against the experimental data collected from the lab-scale studies. The main objective of the modelling effort was to develop a reliable calibrated model that can be utilized to predict performance for HS wastewater, with and without RBF, and assess various scenarios not tested experimentally in this work. Simulations were conducted using COD state influent model, which was used for influent fractionation with COD,

TSS, and TN as measured input variables. TSS/COD coefficients were modified to approximate experimental data for VSS and SCOD. The values of the calibrated parameters and model calculated parameters for the pseudo-steady state are included in Table C4-1 (see Appendix C). The model calculated parameters, VSS, sCOD, TN, and TN, were comparable with the experimental measured values (Table 5-1), which validates the influent specifications.

Fig. 5-4 compares the averages of the steady-state effluent quality parameters including TSS, sCOD, NH_4-N, and NO_x-N in the three SBRs. For all effluent parameters the model predictions were within the range of the average of experimental data +/- the standard deviation. Similarly, Fig. 5-5 compares the MLSS and MLVSS concentrations in the three SBRs. It is clear from the graph that the experimental data matched well with the model with minimal percent differences ranging from 1% to 4%.

The model-based analysis was further extended to evaluate the scenario of a HS RWW, and in turn its impact on nitrogen removal in the SBRs. The reason for evaluating the HS wastewater is that the TSS removal efficiency of the RBF increases with influent TSS due to cake-formation [Franchi and Santoro, 2015] and thus under this scenario the RBF effluent would have the lowest COD/TN ratio and represent the worst-case scenario for conventional BNR processes. The characteristics of the HS RWW were generated using the fractionation of the MS RWW collected in this study as described in the Material and Methods sections. The simulated effluent characteristics of the two HS RWW cases, with and without RBF treatment, are summarized in Table 5-6. It is evident that the effluent quality for the two-simulated HS RWW runs is comparable to the three experimental runs with RWW, PC, and RBF (Table 5-3). The SBRs achieved 97% TSS removal, 92%-94% TCOD removal, and 74%-79% TN removal. The MLSS concentrations in the two-simulated runs indicate that the RBF (operating at high TSS removal efficiency) reduces the secondary clarifier solids loading by 59% compared to the no-primary (i.e., HS RWW). It is also interesting to compare the HS-RBF SBR with the PC SBR since the influent characteristics to both SBRs with respect to COD, nitrogen, and TSS were comparable. It can be concluded that for the RBF technology, within TSS removal of 27%-70%, representing the limits of performance, BNR is not adversely impacted.

Table 5-6. Effluent characteristics of the simulated SBRs

Parameters	Units	HS-RWW SBR	HS-RBF SBR
TSS	mg/l	17	5
VSS	mg/l	12	4
TCOD	mg/l	53	41
SCOD	mg/l	35	35
TN	mg/l	13	13
TKN	mg/l	3	2
NH3	mg/l	1.0	0.7
NO3	mg/l	10.0	10.7
TP	mg/l	3.1	5.3
MLSS	mg/l	4490	1820
MLVSS	mg/l	3180	1410
Alkalinity	mg$CaCO_3$/L	200	237
Sludge production			
Primary sludge production	mg TSS/d	n/a	980
Biological sludge production	mg TSS/d	898	364
Overall sludge production	mg TSS/d	898	1344

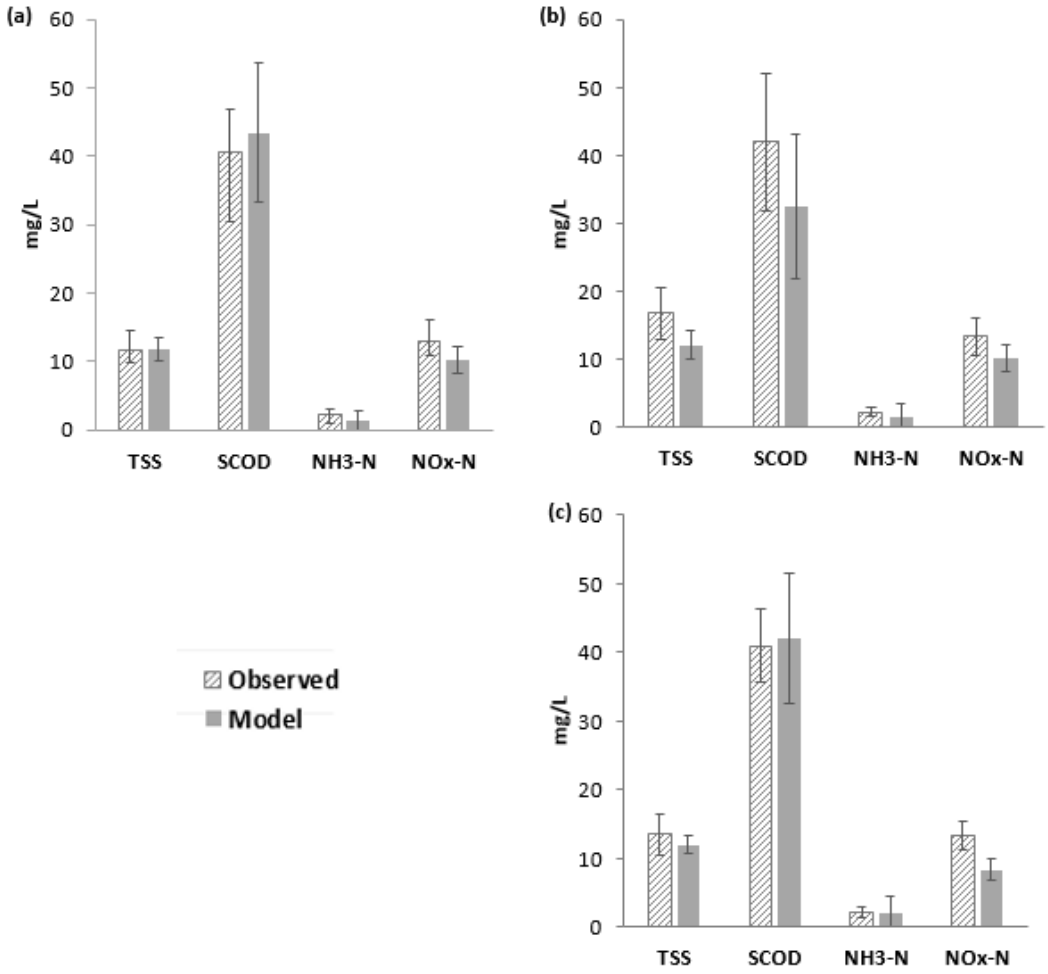

Figure 5-4. Comparison of experimental and modelled average effluent quality for (a) RWW SBR, (b) PC SBR, (c) RBF SBR

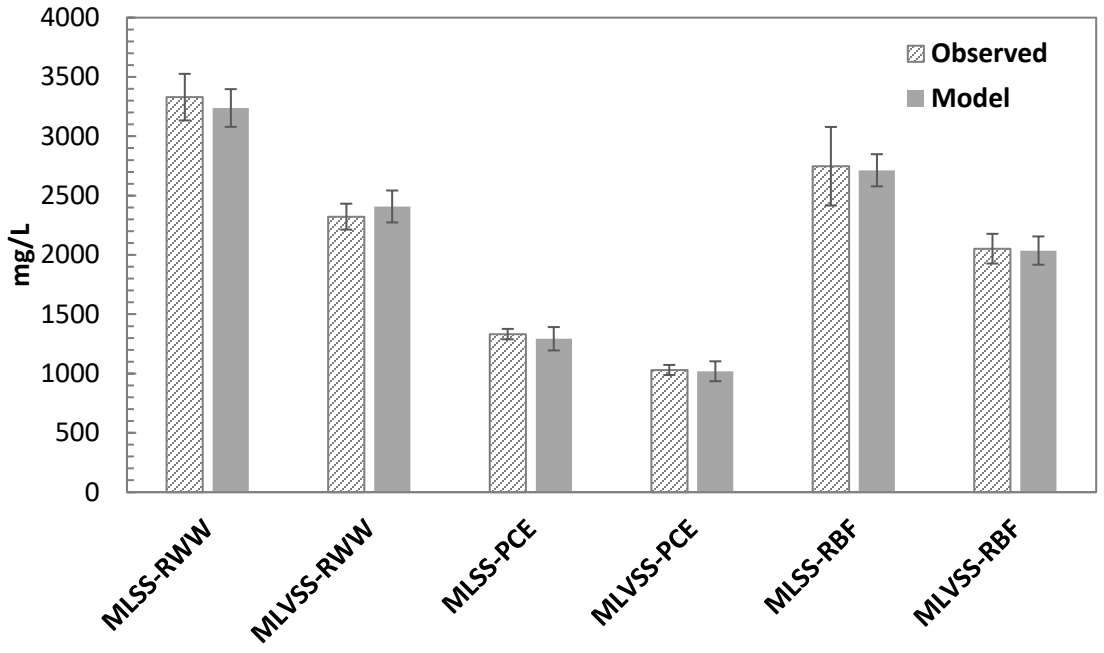

Figure 5-5. Comparison of experimental and modelled average MLSS and MLVSS for the three SBRs

5.4 Conclusions

In this study, the impact of primary microsieving on nitrogen removal was compared against the case of primary clarification, and no primary treatment. Experimental and modelling data obtained from lab-scale SBR reactors suggested that:

1. Despite higher influent TN for RWW SBR and RBF SBR compared to primary clarifier effluent, effluent TN was the same in all three SBRs corresponding to overall TN removal of 73%, 68%, and 71% for RWW, PC and RBF, respectively, indicating that the COD present in the three wastewaters is sufficient for nitrogen removal.
2. Overall sludge production by wastewater treatment plant employing primary treatment by RBF was found to be 9% lower than the primary clarifier, despite its higher biomass yield. Specifically, the observed biomass yields for secondary

sludge was 0.23, 0.20 and 0.29 mg VSS/mg $COD_{consumed}$ for RWW, PC, and RBF SBRs, respectively.

3. Although SDNRs were not significantly impacted by primary treatment, SNR increased with for primary sedimented wastewater.

In light of the above findings, an RBFs offers an alternative level of treatment (to primary sedimentation), which selectively removes particulate solids only, without impacting nitrification and denitrification processes to the extent that is normally observed with primary clarification. It therefore can reduce loads on final sedimentation, while maintaining denitrification capacity, but is less suitable to cases where aeration is limiting, as it does not remove readily degradable organics as effectively as primary sedimentation can.

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Chapter 6

6 Evaluation of Chemically-Enhanced Microsieving on Nitrogen Removal in Wastewater Resource Recovery Facilities

6.1 Introduction

There has been an increased interest and development in alternative carbon diversion technologies in wastewater treatment to increase treatment capacity, especially in geographies with limited space for expansion. Microsieving technology like the rotating belt filters (RBFs) has emerged as a promising primary solids separation alternative to primary clarification with a number of pilot and full-scale installations in operation [Franchi and Santoro, 2015; Ghasimi, 2016; Rusten et al., 2017; Lema and Martinez, 2017]. The RBF requires minimal space and facilitates performance to achieve total suspended solids (TSS) and chemical oxygen demand (COD) removals suitable for downstream biological nutrient removal processes. Furthermore, like chemically-enhanced primary treatment (CEPT) with clarification, microsieving is also emerging into enhanced primary treatment landscape [Rusten et al., 2017; Väänänen, 2017].

CEPT with clarification has been practiced for >50 years all around the world, targeting suspended solids removal, phosphorous (P) removal, reduction in sludge volume, increasing biogas production, treating wet-weather flows, etc. [Parker et al. 2001; CH2M Hill, 2007; He et al., 2016; Kooijman et al., 2017]. CEPT has been successfully integrated ahead of biological treatment at full-scale plants to achieve nitrification and phosphorous removal, whereas in order to achieve nitrogen removal (via denitrification), carbon supplementation is incorporated [Parker et al. 2001]. On the other hand, microsieving technologies with chemical pre-treatment have recently been studied for phosphorous removal, carbon removal to recover energy from sludge digestion, global warming potential, etc. [Remy et al., 2014; Väänänen et al., 2016; Rusten et al., 2017]. Remy et al. [2014] observed 86% TP removal, 70%-80% COD removal, and >95% TSS removal with flocculation and microsieving using drum filter (100 µm). Additionally, the above-mentioned authors who conducted modeling assessment of the global warming

potential of the chemically enhanced microsieving (flocculation, microsieving through 100 µm, biofilter for nitrification/denitrification) process and compared with conventional treatment plant (primary clarifier, activated sludge, nitrification/denitrification, chemical P removal), reported the flocculation-microsieving to be CO_2-neutral (-0.06 vs +0.27 kg CO_2-eq/m^3). Väänänen et al. [2016] reported 80%-90% TSS, 70%-90% COD, and 50%-90% total phosphorous (TP) removal using polymer addition (2-4 mg/L) prior to microsieving (drum filter, 100 µm). Similarly, Rusten et al. [2017] observed 60%-70% TSS removal with chemically enhanced RBF (~1 mg/L polymer).

While there is enough evidence of phosphorous and TSS removal using chemically enhanced RBF, no study has been identified in the literature studying the impact of chemical pre-treatment ahead of RBF on biological nitrogen removal. Additionally, it is essential to compare chemically-enhanced-RBF with RBF-alone, primary clarification, and no-primary, on BNR performance. Therefore, the main objective of this study was to investigate the impact of chemically enhanced RBF on biological nitrogen removal. Four scenarios, no primary treatment (RWW), RBF, chemically-enhanced RBF (CE-RBF), and primary clarification (PC) were compared in four sequential batch reactors (SBRs) operated in a pre-anoxic mode for nitrification and denitrification.

6.2 Materials and Methods

6.2.1 Sample Collection and Preparation

Raw wastewater (RWW) (screened and degritted) and primary clarifier effluent (PC) (retention time of 2 h average flow) was collected twice a week (Nov 2017 to Jan 2018) from the Pottersburg wastewater treatment plant (WTTP) in London, ON, Canada. Laboratory simulation of the RBF effluent was achieved by microsieving a sufficient volume of wastewater through 350 µm (identical to the one used in commercially available full-scale RBF) microsieve to emulate operation at a high hydraulic loading rate to avert cake formation in the full-scale system. The 350 µm microsieve simultaneously optimizes filter capacity and solids retention. This pore size also corresponds to the most widely used microsieve in full-scale applications [Rusten et al., 2017]. Chemically

enhanced-RBF effluent (CE-RBF) was simulated by adding 10 mg/L ferric chloride (as Fe^{3+}) followed by 2 mg/L cationic (polyacrylamide family with 40% active solids) polymer (Part No. PG906, ChemTreat, Virginia, USA), and thereafter sieving through 350 μm mesh. The wastewater samples were stored at 4 ^{o}C until use, which occurred within 72-96 hours. The influent characteristics of the four SBRs, RWW SBR, RBF SBR, CE-RBF SBR, and PC SBR, are provided in Table 6-1.

Table 6-1. Influent characteristics of the four SBRS fed with Pottersburg WWTP wastewater

Parameter	Unit	RWW SBR*	RBF SBR*	CE-RBF SBR*	PC SBR*
TSS	mg/L	171±31	103±12	23±10	67±16
VSS	mg/L	147±29	81±8	18±8	52±13
TCOD	mg/L	434±91	290±45	141±30	225±43
SCOD	mg/L	144±31	121±27	111±22	125±31
TN	mg/L	55±8	52±6	47±6	41±6
NO_3-N	mg/L	0.74±0.61	0.12±0.14	0.06±0.12	0.54±0.64
NO_2-N	mg/L	0.07±0.07	0.06±0.12	0.05±0.08	0.09±0.08
NH_4^+-N	mg/L	30±6	31±6	31±6	24±4
TP	mg/L	5±0.9	5±0.9	1.0±0.4	3±1
Alkalinity	mg $CaCO_3$/L	297±26	293±24	267±21	282±23
pH		7.6±0.3	7.6±0.3	7.4±0.4	7.5±0.3
TCOD/TN		8±2	6±1	3±1	6±1
SCOD/TN		3±0.5	2±0.5	2±0.6	3±1

*Averages and standard deviations of 14 sets of samples

6.2.2 SBR Set-Up and Operation

Four laboratory-scale anoxic-aerobic SBRs with a working volume of 2 L were seeded with sludge from the nitrifying Pottersburg WWTP. The SBRs were operated with a cycle time of 6 h, that is, four cycles per day, at room temperature (22-24 ^{o}C). Each cycle consisted of 10 min anoxic fill, a 1.25 h anoxic react period and a 3.5 h aerobic period (DO ~ 3-4 mg/L), followed by 1 h settling and 0.25 h decanting. A fill ratio of 0.35 (i.e., V_{fill}/V_{toal}) was used. Hence, residual nitrate from the 0.65 fill was removed by organics in the fresh feed, with some nitrate discharged in the decant period. The MLSS wasting rate was 0.2 L/d to maintain a solids retention time (SRT) of approximately 10 d.

Cyclic study with measurements of liquid-phase components was carried out during the steady-state operation to monitor the performance of the SBRs as well as to determine the specific nitrification and denitrification rates. Aliquots (10 mL in volume) were withdrawn at predetermined time intervals and were analyzed for the following parameters: total COD (TCOD); soluble COD (SCOD); NH_4-N; NO_3-N; NO_2-N; soluble phosphorus.

6.2.3 Monitoring, Sampling, and Analysis

The dissolved oxygen (DO) and pH in each SBR were measured with a DO probe (HACH, Canada) and a pH probe (VWR, Canada), respectively. Mixed liquor samples were collected from the SBR periodically and analyzed for total suspended solids (TSS), volatile suspended solids (VSS), and total nitrogen. Influent and effluent samples were collected from the SBR periodically and were analyzed for inorganic nitrogen species (ammonia nitrogen, NH_4-N; nitrate nitrogen, NO_3-N; nitrite nitrogen, NO_2-N), total nitrogen (TN); total phosphorus, (TP); soluble phosphorous; soluble chemical oxygen demand (SCOD); total chemical oxygen demand (TCOD) using Hach test kits (HACH, London, Ontario). Additionally, the influent and effluent samples were also analyzed for total suspended solids (TSS), volatile suspended solids (VSS), and alkalinity based on Standard Methods [APHA, 1998].

6.3 Results and Discussion

6.3.1 Primary Treatment Performance

The TSS removal efficiency (Table 6-1, Row 1) for the RBF ranged from 16% to 54% (averaged 38%) and that of CE-RBF ranged from 73% to 93% (averaged 86%), while that of primary clarification ranged from 26% to 74% (averaged 59%). Rusten et al. [2017] observed similar TSS removal by the RBF, ranging from 35%-45% for influent TSS ranging from 170-270 mg/L at similar operating conditions (no cake formation). Moreover, the above-mentioned authors observed up to 70% TSS removal with a polymer (low cationic charge, high molecular weight polyacrylamide) dose of 1.77 mg/L

in the CE-RBF, which was lower than the ones reported in this study. This was probably because this study used polymer (2 mg/L) plus $FeCl_3$ (10 mg/L). Väänänen [2016] observed >80% TSS removal with CE-RBF (cationic polymer; 100 µm). Other high-rate primary processes such as Actiflo® and DensaDeg® also utilize coagulants and polymers and have reported TSS removal efficiencies ranging from 74% to 92% which are in line with CE-RBF performance in this study [Lema and Martinez, 2017].

Upon comparison with the Greenway study (Chapter 5), it is observed that the TSS removal efficiency by the RBF in this study (38%) was higher compared to the Greenway (27%) plant (Section 5.3.1). Interestingly, the Greenway raw wastewater had much higher influent TSS (330 mg/L; Table 5-1) compared to Pottersburg (171 mg/L; Table 6-1). Franchi and Santoro [2015], and Rusten et al. [2017] both have reported that the TSS removal by the RBF increases with increasing influent TSS concentration, which is inconsistent with the Pottersburg and Greenway data, clearly suggesting that besides the influent TSS concentration, the particle size distribution is another important parameter that governs the TSS removal efficiency of the RBF along with other operating parameters of the RBF including sieve opening, sieve rate, water level, and belt speed. For instance, Razafimanantsoa et al. [2014b] observed TSS removal efficiency of 27% using a 150 µm-sieve (influent TSS of 383 mg/L) and 65% using a 18 µm-sieve (influent TSS of 321 mg/L). As wastewater quality varies from day-to-day and from site-to-site, as evidenced by the range of efficiencies observed at both Pottersburg and Greenway WWTP, in order to achieve the desired removal efficiency by the RBF, a thorough characterization of the wastewater suspended solids particle size distribution is crucial.

Additionally, it was observed that the RBF removed up to 7% TN, 15% TP, and 32% TCOD, while the CE-RBF removed 14% TN, 82% TP, and 67% TCOD. The PC removed 25% TN, 35% TP, and 47% TCOD. Razafimanantsoa et al. [2014b] observed similar TN removal ranging from 5% (150 µm-sieve) to 15% (33 µm-sieve) and TCOD removal ranging from 25% (150 µm-sieve) to 46% (18 µm-sieve). Rusten et al. [2017] observed 19% to 24% TCOD removal with RBF, and Väänänen et al. [2017] observed 70%-95% TCOD removal with CE-RBF (cationic polymer dose of 1-5 mg/L, Fe_3^+ dose of 10-30 mg/L, and 100 µm). Similarly, Väänänen et al. [2017] reported >95% TP

removal with CE-RBF. Moreover, it is well known that chemically enhanced primary clarification, including high-rate processes like Actiflo® and DensaDeg®, can achieve 75%-95% TP removal [Tchobanoglous et al., 2003; Lema and Martinez, 2017], thus, the TP removal in CE-RBF is justified. Furthermore, the % particulate N, P, COD removal was plotted against the VSS removal (Fig. 6-1), and it was observed that the removal of particulate N, P, and COD increased with VSS removal. Hence, the CE-RBF removed 56%, 79%, and 89%, whereas the RBF removed 18%, 18%, and 41% of the particulate N, P, and COD, respectively. Similarly, the PC removed, 37%, 52%, and 65% of the particulate N, P, and COD, respectively.

The TCOD/TN ratio in the four wastewaters was different (Table 6-1). The RWW had the highest ratio of 8 (ranging from 6-11), compared to RBF at 6 (ranging from 5-7), CE-RBF at 3 (ranging from 2-5), and PC at 6 (ranging from 4-8). Interestingly, the CE-RBF treatment led to SCOD removal of 23% (ranging from 5% to 40%) which suggests that the polymer-$FeCl_3$ chemical addition led to adsorption of SCOD. The 23% reduction was statistically significant at the 95 percent confidence interval. The corresponding SCOD/TN ratio were 3, 2, 2, and 3 for RWW, RBF, CE-RBF, and PC, respectively. Remy et al. [2014] observed similar ~30% removal of soluble COD from raw wastewater by coagulation (polyaluminium chloride, 15-20 mg/L)-flocculation (polymer, 5-7 mg/L)-microsieving with drum filter (Hydrotech, 100 μm).

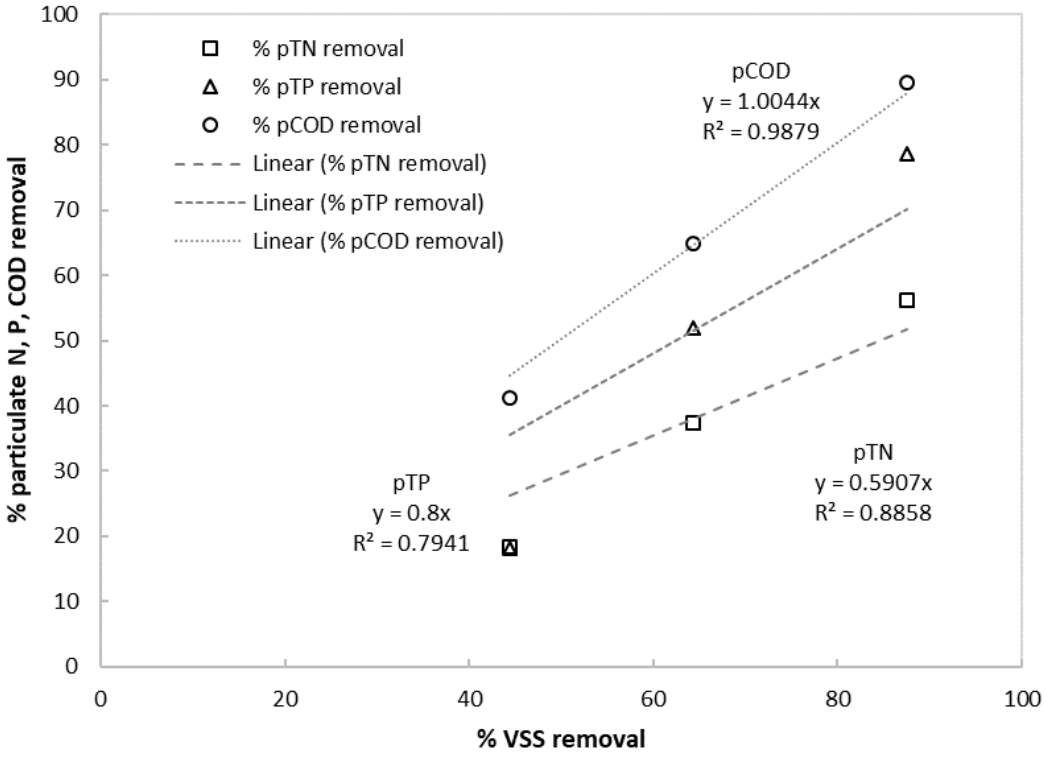

Figure 6-1. Particulate nitrogen, phosphorous, and COD removal against VSS removal

6.3.2 SBR Performance

The temporal variations of SBR influent and effluent TSS and COD are presented in Fig. 6-2a and 6-2b, respectively. Effluent characteristics of the four SBRs during steady-state operation are summarized in Table 6-2. TSS removal efficiencies of 94%, 86%, 63%, and 87% was observed for the RWW, RBF, CE-RBF, and PC-SBRs, respectively with average effluent TSS concentration of 10±3 mg/L. Razafimanantsoa et al. [2014b] observed similar TSS (76%-95%) removal efficiencies in SBRs fed with RWW, 18, 33, 50, 90, and 150 µm-sieved wastewater. The lower TSS removal efficiency in the CE-RBF SBR was not due to poor settleability in the SBR, but due to the low influent TSS (23 mg/L) itself. Similarly, the four SBRs achieved good TCOD removal efficiencies, averaging 92%, 88%, 85%, and 90% for the RWW, RBF, CE-RBF, and PC-SBRs, respectively. Razafimanantsoa et al. [2014b], Rusten et al. [2016], and Greenway study

(Chapter 5) observed similar TCOD (>84%) removal efficiencies in SBRs fed with RWW, 18, 33, 50, 90, and 150 µm-sieved wastewater.

Table 6-2. Effluent characteristics of the four SBRS fed with Pottersburg WWTP wastewater

Parameter	Unit	RWW SBR*	RBF SBR*	CE-RBF SBR*	PC SBR*
TSS	mg/L	10±1	14±4	9±2	9±3
VSS	mg/L	8±1	10±3	6±2	6±3
TCOD	mg/L	37±10	35±5	21±4	23±6
SCOD	mg/L	30±11	24±7	16±4	17±6
TN	mg/L	25±5	28±7	33±4	29±5
NO_3-N	mg/L	20±5	24±8	26±5	24±5
NO_2-N	mg/L	0.11±0.06	0.08±0.05	0.03±0.02	0.04±0.02
NH_4^+-N	mg/L	2.3±0.5	2.2±0.4	2.3±0.5	2.3±0.4
TP	mg/L	4±0.7	3±0.8	0.6±0.1	3±0.6
Alkalinity	mg $CaCO_3$/L	142±16	122±22	93±9	131±11
pH		7.5±0.3	7.6±0.2	7.6±0.6	7.7±0.1

*Averages and standard deviations of 11 sets of samples

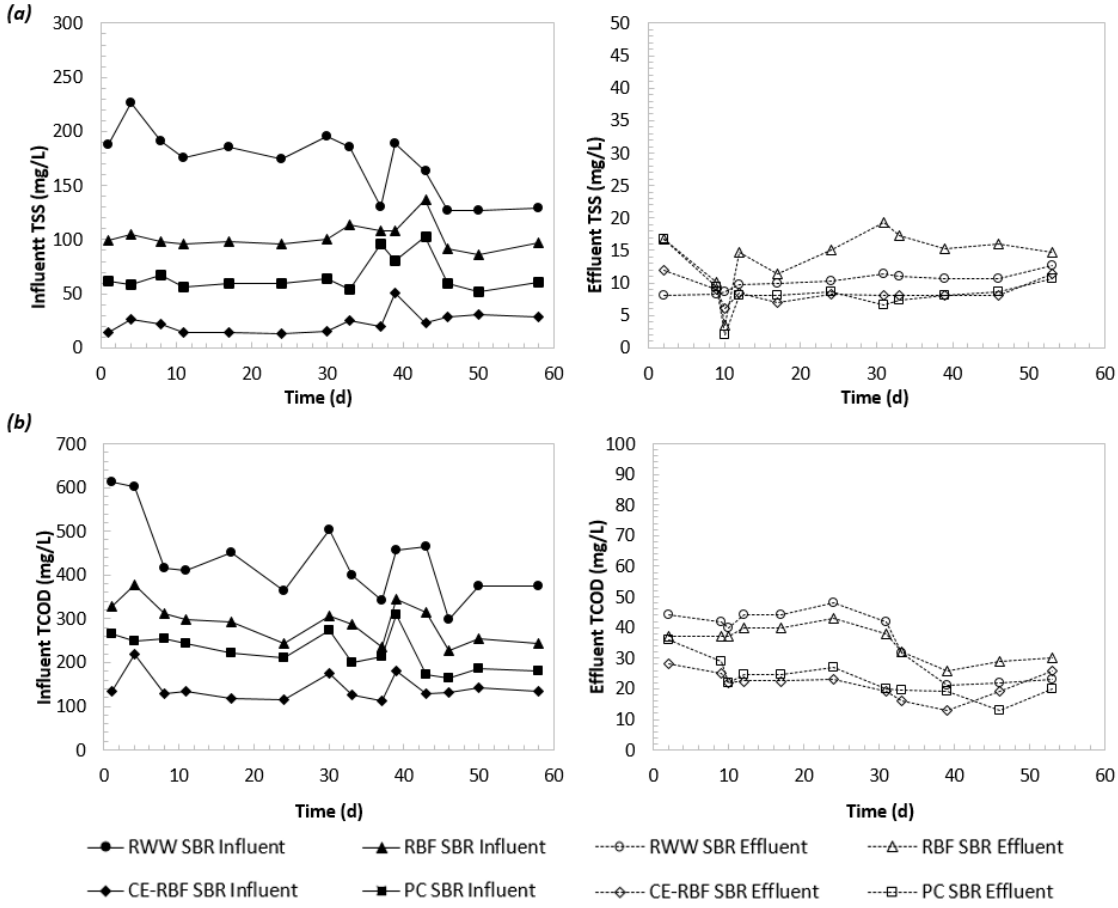

Figure 6-2. SBR performance: Influent and effluent (a) TSS; (b) COD concentrations

Fig. 6-3a, 6.3b, and 6.3c illustrate the experimental results for the TN and species throughout the duration of the experiment. Effluent characteristics of the four SBRs during steady-state operation are also summarized in Table 6-2. Unlike the Greenway SBRs study, the effluent TN in the four SBRs was different. Influent ammonia concentration ranged in the influent from 24 to 31 mg NH_4^+-N/L whereas the concentration in the effluent for all four SRBS averaged at 2±0.05 mg NH_4^+-N/L, corresponding to ≥90% nitrification efficiency. The effluent ammonia (Fig. 6-3c) was consistent with the Greenway study (~2 mg NH_4^+-N/L) and meets the site-specific limits (3-10 mg/L NH_4^+-N) in Ontario [Eini et al., 2017]. Nitrites in the influent and effluent were negligible (≤0.1 mg NO_2-N/L). On an average 20 mg NO_3-N/L were observed in the RWW SBR effluent, 24 mg NO_3-N/L were observed in the RBF and PC SBR, and the

highest, 26 mg NO_3-N/L, were observed in the CE-RBF SBR effluent (Fig. 6-3b). While high residual nitrates are an artifact of operating SBRs in pre-anoxic mode, nevertheless, the high residual nitrates in this study were indication of poor nitrogen removal efficiency. Overall TN removal for the four SBRs averaged at 54%, 45%, 30%, and 29% for RWW, RBF, CE-RBF, and PC-SBRs, respectively. As discussed earlier, the TCOD/TN ratio in the four influents was 8, 6, 3, and 6, for RWW, RBF, CE-RBF, and PC-SBRs, respectively, whereas the SCOD/TN ranged from 2-3. Typically, COD/N ratio of 6 to 10 is required for efficient nitrogen removal [Kooijman et al., 2017]. The low SCOD/TN ratios indicate that the SBRs were carbon limited which reduced denitrification capacity. In order to confirm this theory, one of the cycles of the SBRs was switched with sodium acetate feed, which is an ideal carbon source for denitrification (Appendix D). Higher nitrate uptake rates, compared to wastewater feed (discussed in later section) were observed in the RWW, RBF, and PC SBR while negligible uptake was observed in the CE-RBF SBR. This confirmed that the SBRs were carbon limited for denitrification, while the CE-SBR was carbon limited as well as biomass limited. It was verified that the biomass limitation observed in the CE-RBF SBR was not due to phosphorous limitation. Remy et al. [2014] predicted CE-RBF process requiring external carbon source to drive denitrification for nitrogen removal. Razafimanantsoa et al. [2014b] observed TN removal of 55% and 57% in 150 µm-sieved SBR and RWW SBR, respectively, with both having the same SCOD/TN ratio of 4. The SCOD/TN ratio in the Greenway study ranged from 6-7 and accordingly, higher TN removal efficiencies of 71%-73% was observed in the RBF and RWW SBRs. Based on these results, it can be concluded that high TSS removal in the primary treatment step, especially in low-strength wastewater, can lead to decline in nitrogen removal efficiency as the COD present in the wastewater becomes insufficient for nitrogen removal.

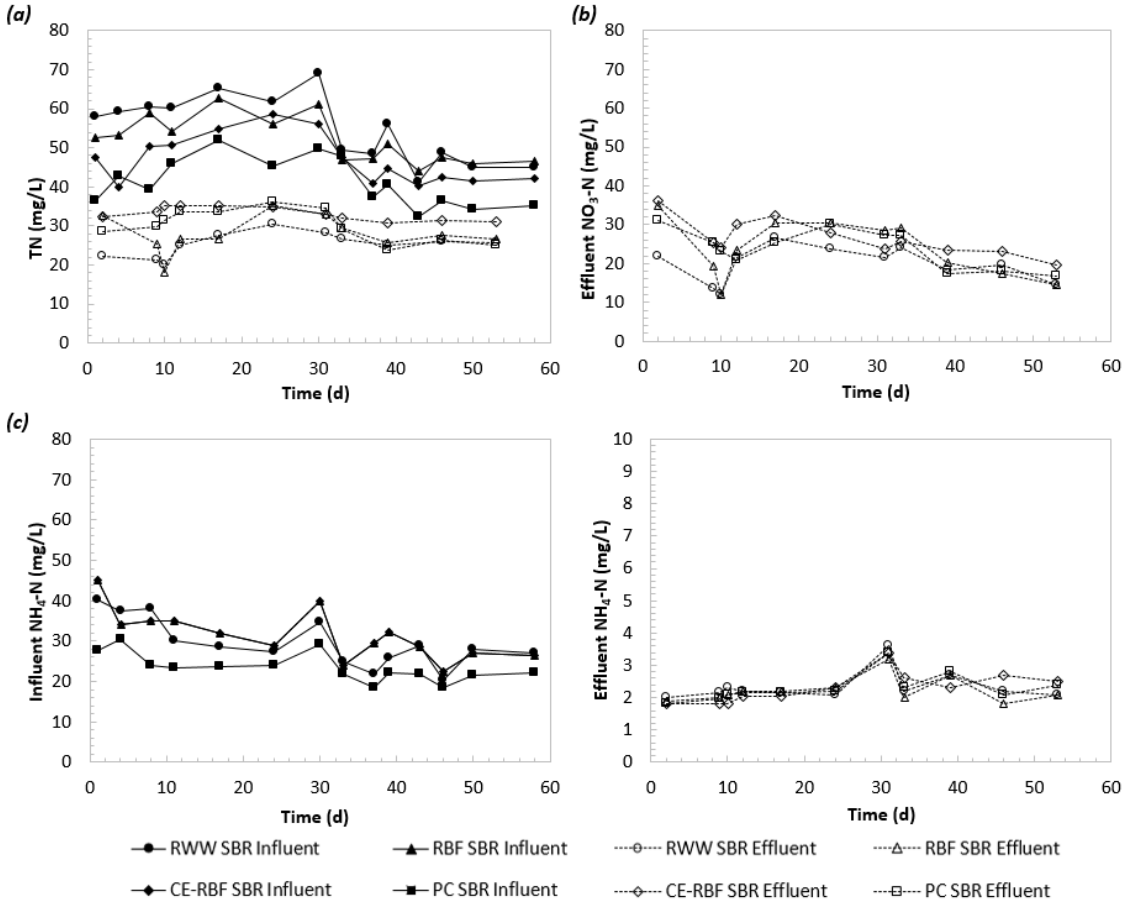

Figure 6-3. SBR performance: (a) Influent and effluent TN; (b) residual NO_3-N in the effluent; and (c) Influent and effluent NH_4-N

6.3.3 Sludge Production and Biomass Yield

The steady-state concentrations of MLSS in the four SBRs were observed to be 3114, 1790, 846, and 1538 mg/L for the RWW, RBF, CE-RBF, and PC-SBRs, respectively (Table 6-3). The MLSS concentrations suggest that the RBF and the PC reduce the secondary clarifier solids loading by 43% and 51%, respectively, whereas the CE-RBF reduces it significantly by 73%. The reduction in secondary clarifier loading with Pottersburg PC (this study) was comparable with the Greenway PC (Chapter 5). Additionally, the TSS removal efficiency of the PC's at the two plants was comparable (63%±5%). It is important to acknowledge the obvious trend in percent reduction in secondary clarifier loading with respect to TSS removal in the primary step. Fig. 6-4 includes the results of this study as well as the study on Greenway wastewater (Chapter

5). This trend could likewise be related to the secondary sludge production, where the higher removal of suspended solid in primary treatment, the lower the secondary sludge production. The fraction of non-biodegradable VSS in the influent ($nbVSS_{influent}$) was estimated based on the particulate COD (XCOD) and VSS plot (Fig. 6-5a) and assuming 45% of the XCOD is non-biodegradable (Chapter 4, Table 4-2). The nbVSS would accumulate in the SBRs in accordance to the following Eq. 6.1:

$$nbVSS\ accumulation\ \left(\frac{mg}{L}\right) = \left(\frac{SRT}{HRT}\right)\left(nbVSS_{influent}\right) \quad (6.1)$$

The nbVSS was observed to be 29%, 30%, 11%, and 21% of the MLSS measured in the RWW, RBF, CE-RBF, and PC SBR, respectively. Additionally, the nbVSS accumulation was correlated with the inert suspended solids (ISS) in the influent for both Pottersburg and Greenway SBRs (Fig. 6-5b). Similarly, the accumulation of the ISS (from influent) accounted for 11%, 17%, 8%, and 13% of the MLSS in the SBRs. Overall accumulation of nonbiodegradable volatile and non-volatile solids were 44%, 46%, 20%, and 34% of the MLSS. Thus, the direct correlation of the reduction in secondary clarifiers solids loading with the primary treatment SS removal efficiency is rationalized by the high contribution of nonbiodegradable SS to the overall biosolids production.

The observed biomass yields from the linear fits of cumulative VSS production versus COD removed were 0.30, 0.29, 0.25, 0.26 mg VSS/ mg $COD_{consumed}$ for the RWW, RBF, CE-RBF, and PC-SBRs, respectively (Appendix D). The Greenway WWTP study observed higher biomass yield in the RBF SBR (0.29 mg VSS/ mg $COD_{consumed}$) than the RWW SBR, (0.23 mg VSS/ mg $COD_{consumed}$), however, in this study the yields in the RWW and RBF-SBR were comparable. Razafimanantsoa et al. [2014b] observed biomass yield of 0.27-0.29 mg VSS/mg COD in RBF SBR (90 µm and 150 µm-sieved) compared to RWW SBR (0.21 mg VSS/mg COD). The volatile fraction of MLSS in RWW (0.74) and RBF SBRs (0.70) was observed to be higher than the CE-RBF SBR (0.64) and the PC SBR (0.67). The above-mentioned results were in contrast to the Greenway study, where the volatile fraction in PC SBR (0.76) was higher compared to the RWW (0.70) and RBF (0.73) SBRs. It is also interesting to observe that the trend of

the biomass yield in the four SBRs was similar to the % nbVSS of the MLSS, emphasizing the important role of primary treatment in removing inert suspended solids.

Sludge production was calculated based on the percent TSS removal by respective primary treatment and sludge wastage rates from the SBRs. Although the biological sludge production with primary clarification system (308 mg TSS/d) was 14% lower than the RBF system (358 mg TSS/d), the overall primary and biological sludge produced by the RBF system was 9% lower than the primary clarifier (541 mg TSS/d versus 592 mg TSS/d). Similarly, the biological sludge production by the CE-RBF SBR was 45% lower than the PC SBR, however, the overall sludge production between the two was similar primarily because of high primary sludge produced by the CE-RBF. The Greenway study (Chapter 5) observed the same 9% reduction in overall sludge production when comparing PC and the RBF SBRs. Furthermore, the RBF system achieved 6% lower overall sludge production compared to the CE-RBF SBR (541 mg TSS/d versus 579 mg TSS/d).

Table 6-3. Concentrations MLSS and sludge production

SBR	Unit	RWW SBR	RBF SBR	CE-RBF SBR	PC SBR
MLSS	mg/L	3114±404	1790±455	846±127	1538±162
MLVSS	mg/L	2290±302	1257±323	543±103	1038±135
nbVSS*	mg/L	918	535	95	317
MLVSS/MLSS		0.74	0.70	0.64	0.67
Biomass yield	mg VSS/ mg $COD_{consumed}$	0.30	0.29	0.25	0.26
Sludge production					
Primary sludge production	mg TSS/d	0	183	409	284
Secondary sludge production	mg TSS/d	623	358	169	308
Overall sludge production	mg TSS/d	623	541	579	592

* Estimated based on Eq. 6.1

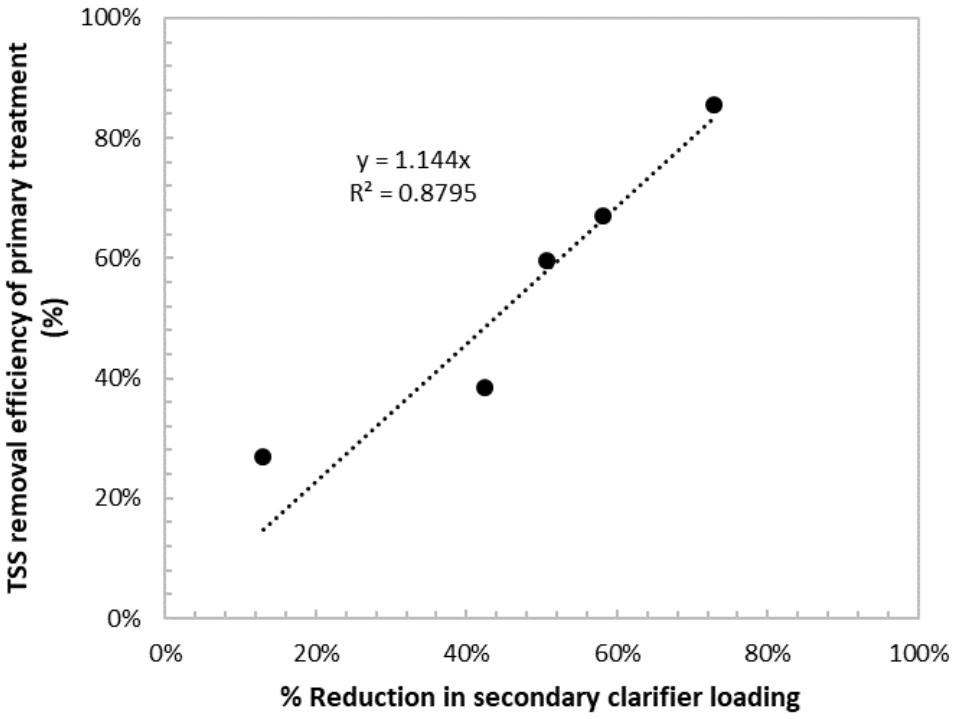

Figure 6-4. TSS removal efficiency versus percent reduction in secondary clarifier loading

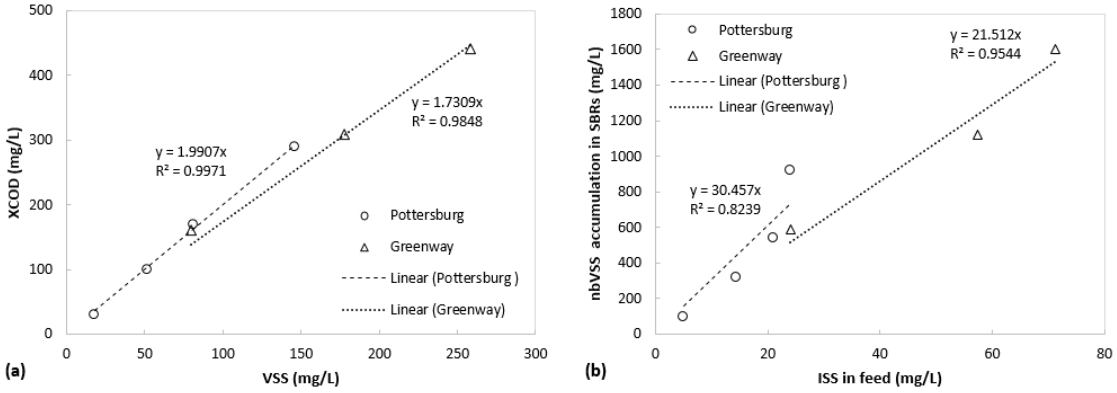

Figure 6-5. (a) XCOD versus VSS; (b) nbVSS accumulation versus ISS in feed for Pottersburg and Greenway SBRs

6.3.4 Steady-State Nitrogen Balance

Nitrogen balances were calculated based on the nitrogen assimilated in the biomass (Eq. 6.2) as well as the nitrogen denitrified (Eq. 6.3) based on influent and effluent TN concentrations.

$$N\ in\ biomass = \left[N\ content\ of\ biomass, \frac{mg\ N}{mg\ MLVSS}\right]\left[Y, \frac{mg\ MLVSS}{mg\ COD_{consumed}}\left(TCOD_{inf} - SCOD_{eff}\right)\right] \quad (6.2)$$

$$N\ denitrified = TN_{inf} - N_{biomass} - TN_{eff} \quad (6.3)$$

As can be seen in Fig. 6-6 and based on the total nitrogen removal of 54% 46%, 29%, and 26% in RWW, RBF, CE-RBF, and PC-SBRs, respectively, the percentages of nitrogen remaining in the effluent were 46%, 54%, 71%, and 73%, respectively. Similarly, nitrogen assimilated in the biomass was 22%, 15%, 7%, and 13% of the influent TN for the RWW, RBF, CE-RBF, and PC-SBRs, respectively. Correspondingly, the amount of oxidizable nitrogen in the four SBRs was comparable at 42±4 mg N/L. Based on Eq. 6.2 and nitrogen mass balance, nitrogen removed by denitrification was 32%, 31%, 23%, and 13% for the RWW, RBF, CE-RBF, and PC-SBRs, respectively, and the amount of COD removed anoxically accounted for 46, 38, 26, and 17 mg COD/L.

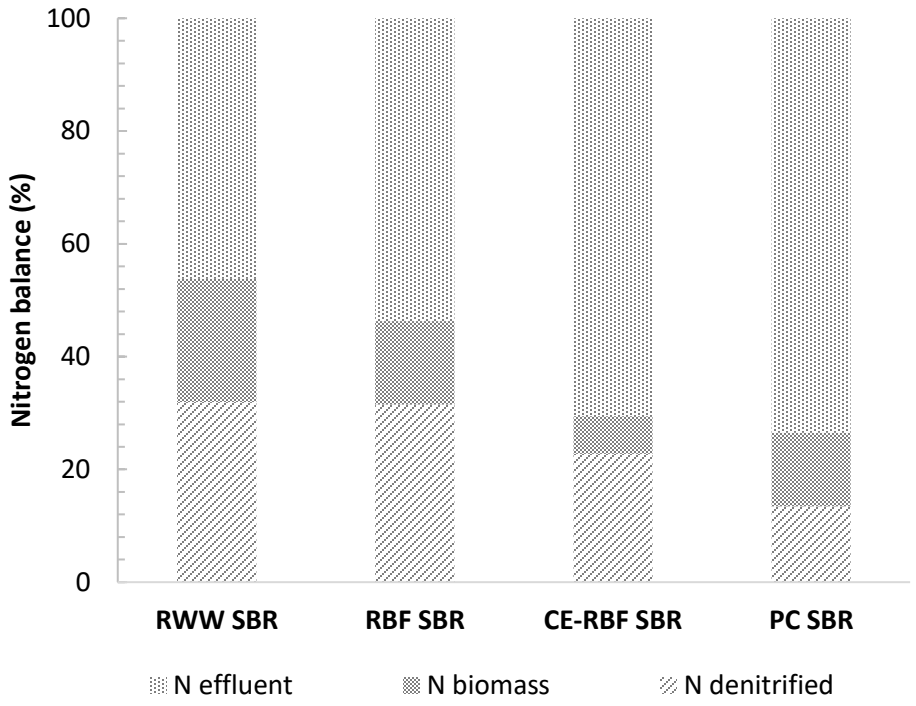

Figure 6-6. Characteristics of nitrogen balance of the four SBRS fed with Pottersburg WWTP wastewater

6.3.5 Nitrification and Denitrification Rates

The specific nitrification rate (SNR) and denitrification rate (SDNR) for the different SBRs are summarized in Table 6-4. The SNRs and SDNRs were calculated based on one cyclic study (profiles in Appendix D) and the average MLVSS measured (also reported in Table 6-3) in the SBRs. The SNRs ranged from 53 to 97 mg NH_3-N/g MLVSS/d in the three SBRs. The SNRs observed in this study were lower in comparison to the ones observed in the Greenway SBRs (ranging from 72-205 mg NH_3-N/g MLVSS/d), however, a similar trend, where the SNRs in CE-RBF and PC SBRs (not RBF) were higher than the RWW SBR, was observed. In other words, the SNRs increased with primary treatment performance (except RBF SBR in this case). Razafimanantsoa et al. [2014b] observed SNRs ranging from 49 to 83 mg NH_3-N/g MLVSS/d for RWW and RBF (50 µm sieve) SBRs, respectively.

The SDNRs observed in this study ranged from 16 to 29 mg NO_3-N/g MLVSS/d and 18-24 mg NO_x-N/g MLVSS/d. Razafimanantsoa et al. [2014b] reported rates in the range of 48-54 mg NO_x-N/g VSS/d which were higher than the rates observed in this study, however the SBRs were operated at TCOD/TN ratios ranging between 9-10 compared to ratios ranging from 3-8 (Table 6-1) in this study. Greenway SBRs which were operating at TCOD/TN ratio of 11 to 13 demonstrated higher rates than this study, ranging from 145 to 196 mg NO_x-N/ g MLVSS/d. The SDNRs determined in this study (22±2 mg NO_x-N/ g MLVSS/d) were comparable, irrespective of the primary treatment, which suggests that the removal of SS from the RWW did not have a significant impact on the rates. However, the denitrification potential itself was limited by the carbon in the raw wastewater (especially CE-RBF SBR which had TCOD/TN ratio of 3) resulting in low overall nitrogen removal. Razafimanantsoa et al. [2014a; 2014b] did not observe differences in the SBRs fed with RWW, 33 µm-sieved, 50 µm-sieved, 90 µm-sieved, and 150µm-sieved wastewater, as well as the Greenway SBRs fed with RWW and 350 µm-sieved wastewater. Additionally, Yan et al. [2017] reported SDNRs of 19 mg NO_x-N/g VSS/d for TCOD/TN ratio of 3.4, which is in line with the observed rate in CE-RBF SBR in this study.

Table 6-4. Specific nitrification and denitrification rates

Rates	**RWW SBR**	**RBF SBR**	**CE-RBF SBR**	**PC SBR**
Specific nitrification rate (mg NH_3-N/ g MLVSS/d)	62	53	74	97
Specific denitrification rate (mg NO_3-N/ g MLVSS/d)	26	29	16	27
Specific denitrification rate (mg NO_x-N/ g MLVSS/d)	21	24	18	21

6.4 Conclusions

In this study, the impact of chemically enhanced microsieving on nitrogen removal was compared against microsieving alone, primary clarification, and no-primary treatment on wastewater with a relatively strength lower than that of Greenway (48% lower TSS and 42% lower TCOD). Experimental results obtained from the lab-scale SBR reactors indicated the following:

1. The four SBRs achieved good TCOD removal efficiencies (>85%).
2. Overall TN removal of 54%, 45%, 30%, and 29% for RWW, RBF, CE-RBF, and PC SBRs was observed. TN removal was carbon limited in all the four SBRs. CE-RBF SBR was observed to be carbon limited as well as biomass limited.
3. The overall sludge production by a wastewater treatment plant employing CE-RBF was similar to the PC. Additionally, the biomass yields for the biological sludge were 0.25 and 0.26 mg VSS/mg $COD_{consumed}$ for CE-RBF and PC SBRs, respectively.
4. The SDNRs were not impacted by the primary treatment, whereas SNRs increased for the CE-RBF and PC SBR.

CE-RBF treatment is ideal for plants trying to achieve BOD and ammonia limits, however, excessive removal of carbon compromises nitrogen removal, especially with low-strength wastewaters. Additionally, the CE-RBF would be an ideal option for carbon diversion in low COD/N utilizing emerging nitrogen removal processes such as anammox.

6.5 References

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Chapter 7

7 Conclusions and Recommendations

7.1 Conclusions

The detailed summary of the major findings of the various subprojects have been included in chapters 3-6. The principal findings of this study were:

I. Of the four methods tested for cellulose determination in wastewater/sludges, including dilute-acid hydrolysis, concentrated acid hydrolysis, enzymatic hydrolysis, and the Schweitzer method, the Schweitzer reagent method was the only reliable method. Schweitzer method does not depend on the hydrolysis efficiency and reduced products analysis, but instead uses a dissolution-extraction method with gravimetric quantification of the precipitate formed. The complete recovery of both standards used i.e., toilet paper and α-cellulose, as well as the relative quickness and ease of the Schweitzer method renders it the most ideal method for cellulose determination in wastewater and sludge samples.

II. The RWW is predominantly biodegradable where 71% of the TCOD was observed to be biodegradable. PC and RBF treatment increased the biodegradable fraction to 78% and 74%, respectively, by removing inert particulates by settling and sieving, respectively. Moreover, microsieving and settling do not impact the soluble components in the wastewaters as reflected by the same S_S, S_I, and S_H, for RWW, PCE, and RBFE.

III. RBFs offers an alternative level of treatment (to primary sedimentation), which selectively removes particulate solids only, without impacting nitrification and denitrification processes to the extent that is normally observed with primary clarification.

    a. A direct correlation of the reduction in secondary clarifiers solids loading with the primary treatment SS removal efficiency was rationalized by the high contribution of nonbiodegradable SS to the overall biosolids

production. The overall primary and biological sludge produced by the RBF was 9% lower than the primary clarifier.

b. CE-RBF treatment is ideal for plants trying to achieve BOD and ammonia limits, however, excessive removal of carbon compromised nitrogen removal, especially with low-strength wastewaters.

7.2 Limitations

Both RBF and the CE-RBF SBRs achieved good TCOD, TSS, and ammonia removal efficiencies with lower or comparable (compared to primary clarifier) overall sludge production. Thus, both scenarios are promising for plants with BOD and ammonia limits. However, the nature of operation of the RBF can impose some limitations. Although the literature suggests that higher influent TSS leads to higher TSS removal efficiency, however, contradictory results were obtained in this study. Therefore, thorough characterization of the wastewater suspended solids particle size distribution is crucial to control RBF performance to better accommodate and control downstream biological processes.

7.3 Recommendations

The successful integration of the RBF as a primary treatment alternative would require further investigation and validation. The following recommendations for future work are made:

I. Cellulose mapping across multiple treatment plants is recommended to study the fate of cellulose at treatment facilities.

II. It is recommended to study the logistics and economics of the Schweitzer's method as a potential method for *recovery* of cellulose fibers from cellulose-rich-RBF sludge.

III. Study the impact of RBF in lab-scale or pilot-scale continuous-flow systems (perhaps MLE configuration) to achieve TN limit of <3 mg/L to overcome residual nitrates due to SBR operation.

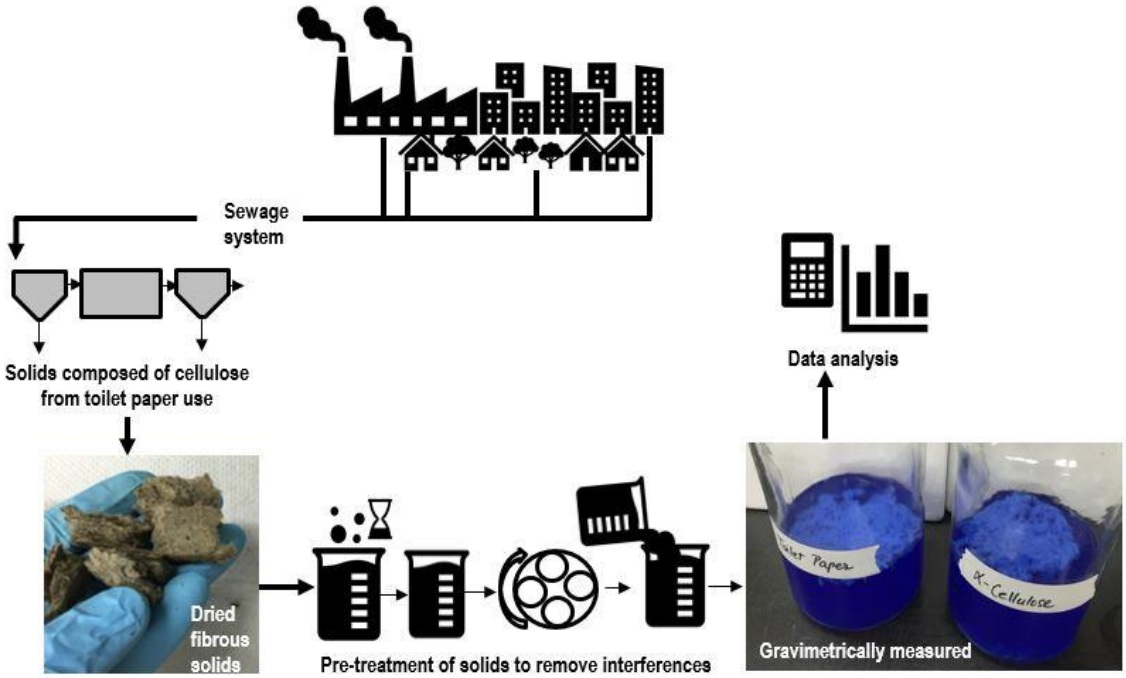
IV. Fermentation of RBF sludge and supplementing fermentate (as a carbon source rich in volatile fatty acids) to feed to secondary biological nitrogen removal processes in order to overcome carbon limitation in CE-RBF treating low-strength wastewater.

V. Study the impact of chemically-enhanced RBF on high-strength municipal wastewater.

VI. Study the impact of RBF in lab-scale or pilot scale continuous-flow systems (perhaps A2O configuration) to achieve enhanced biological phosphorous removal (EBPR).

Appendices

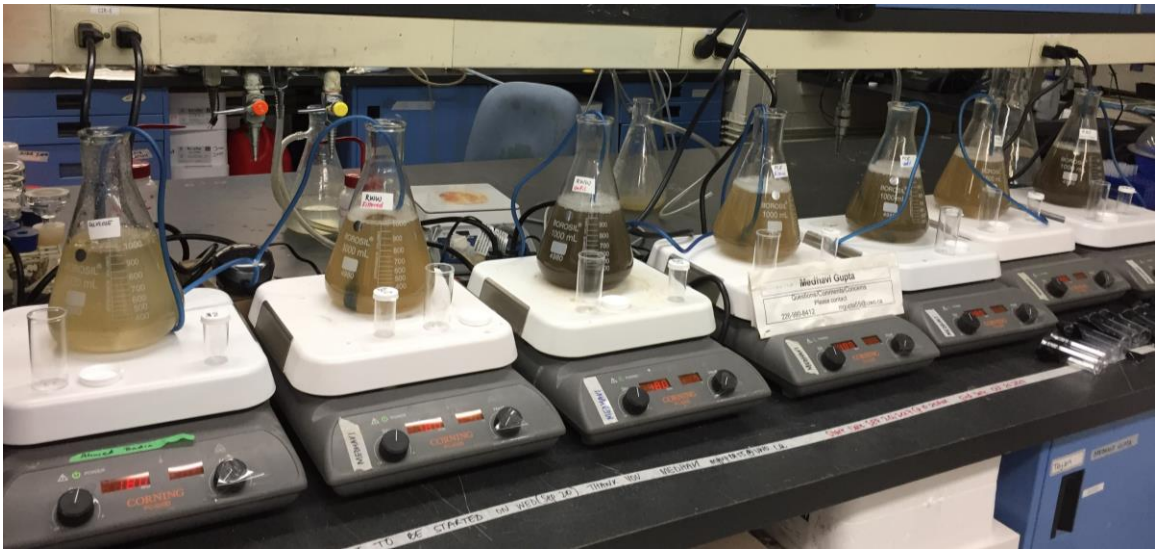
Appendix A. Graphical abstract of Chapter 3



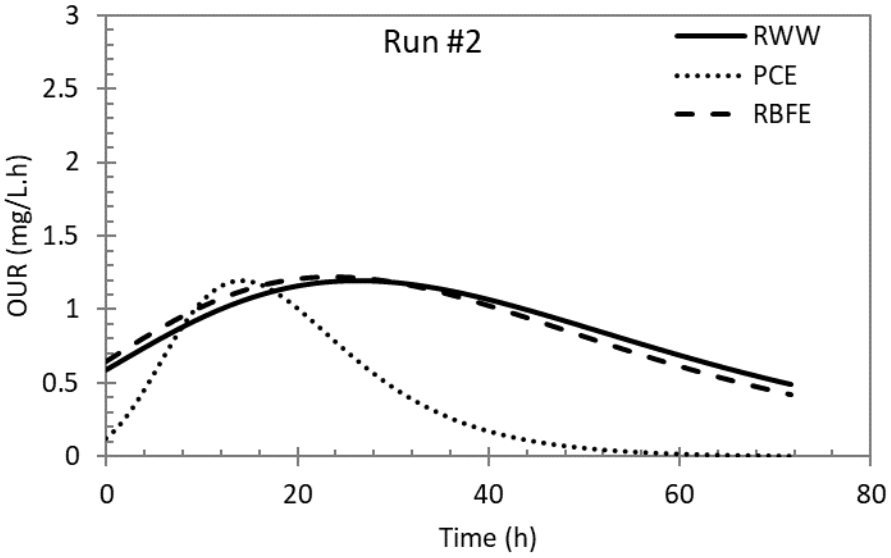
Appendix B. Supplementary material of Chapter 4

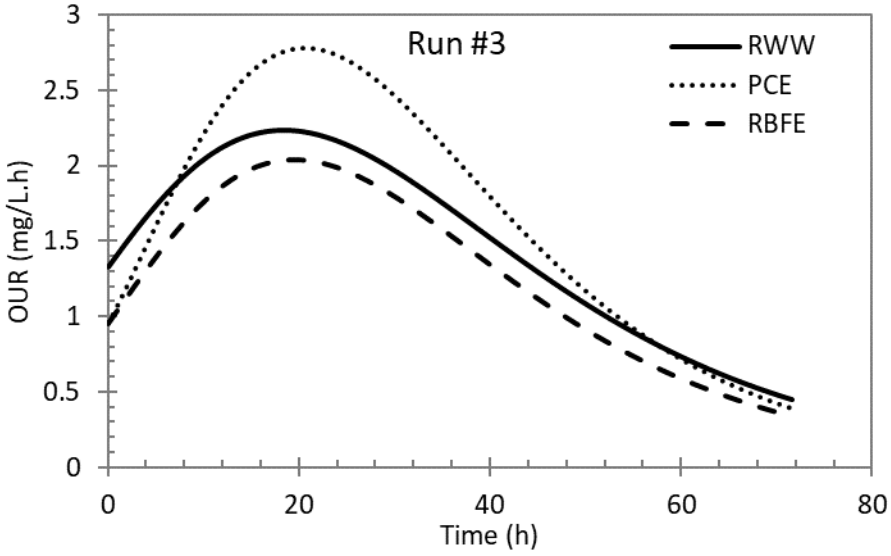
Respirometry set-up

SBR set-up



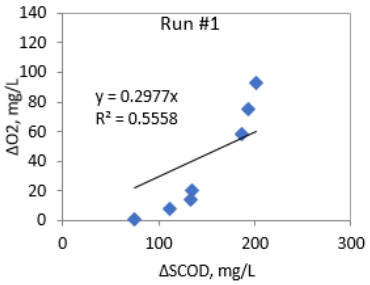
OUR profiles for Run #2 and Run #3



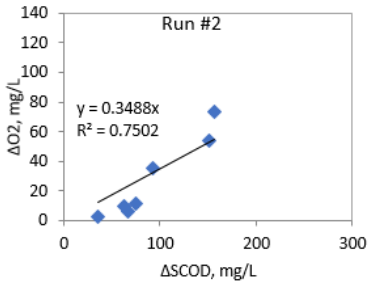


Biomass yield coefficient was calculated based on the equation: $Y_H = 1 - \frac{\Delta O_2}{\Delta SCOD}$

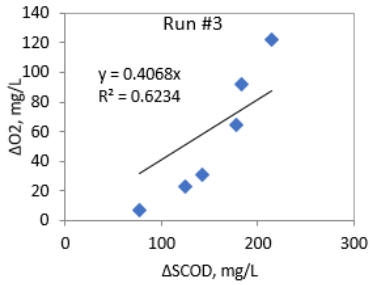
Raw wastewater (RWW)



$Y_H = 1 - 0.2977 = 0.70 \frac{mg\ COD}{mg\ COD}$

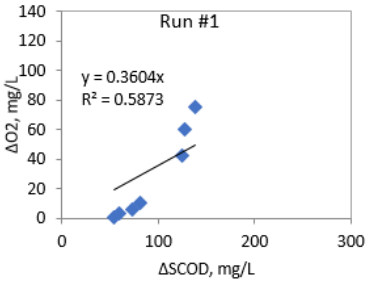


$Y_H = 1 - 0.3488 = 0.65 \frac{mg\ COD}{mg\ COD}$

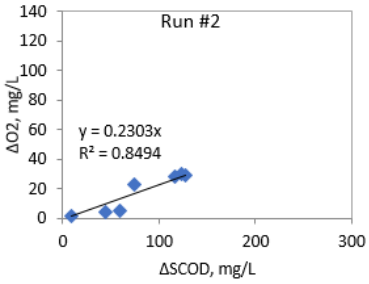


$Y_H = 1 - 0.4068 = 0.59 \frac{mg\ COD}{mg\ COD}$

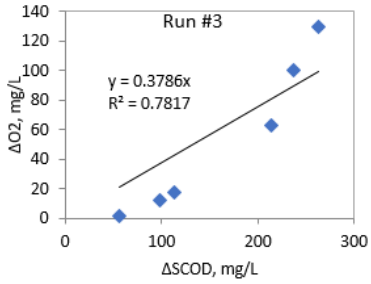
Primary clarifier effluent (PCE)



$Y_H = 1 - 0.3604 = 0.64 \frac{mg\ COD}{mg\ COD}$

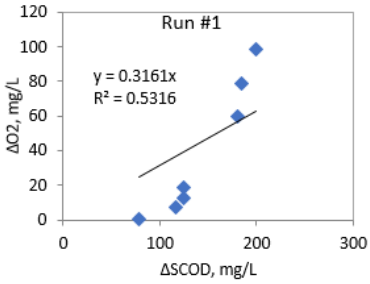


$Y_H = 1 - 0.2303 = 0.77 \frac{mg\ COD}{mg\ COD}$

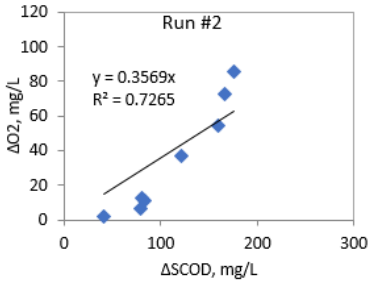


$Y_H = 1 - 0.3786 = 0.62 \frac{mg\ COD}{mg\ COD}$

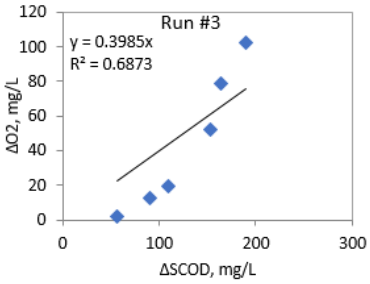
Rotating belt filter effluent (RBFE)



$Y_H = 1 - 0.3161 = 0.68 \frac{mg\ COD}{mg\ COD}$

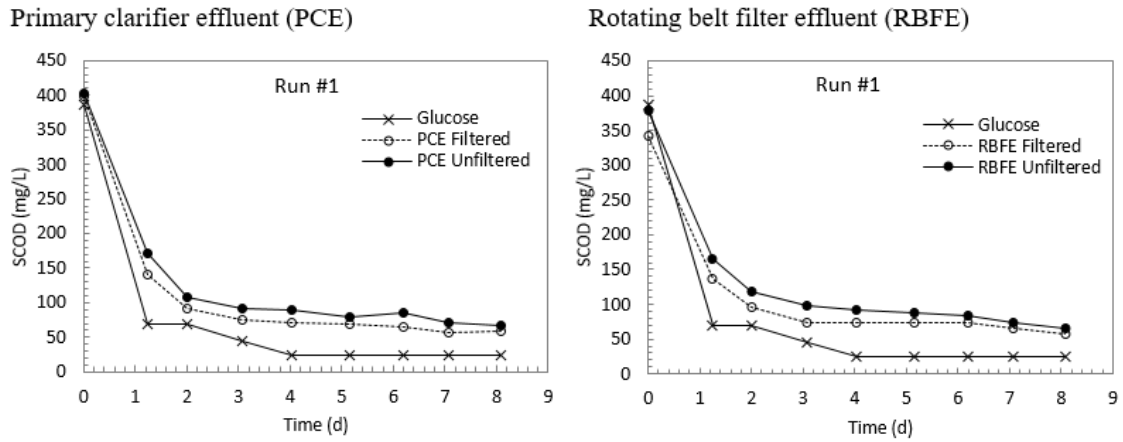


$Y_H = 1 - 0.3569 = 0.64 \frac{mg\ COD}{mg\ COD}$

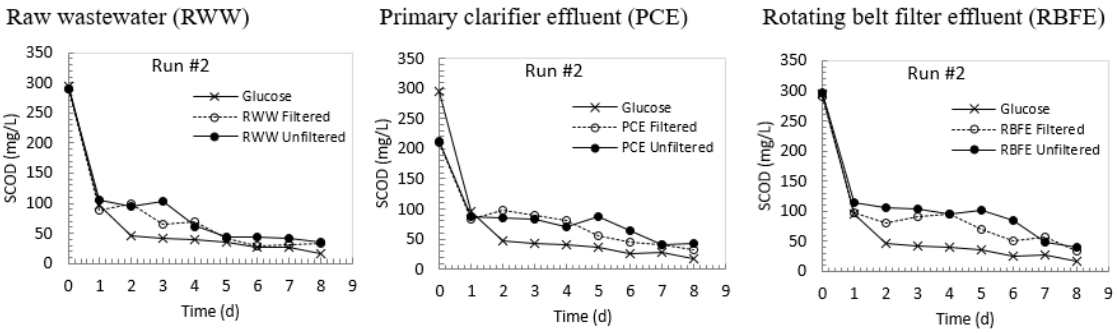


$Y_H = 1 - 0.3985 = 0.60 \frac{mg\ COD}{mg\ COD}$

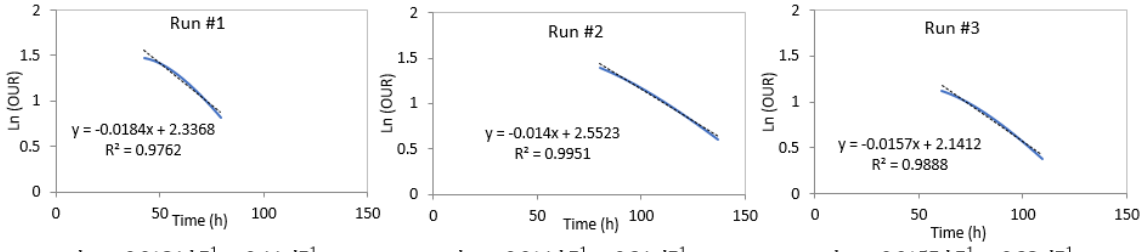
SCOD profiles in the SBRs for Run #1



SCOD profiles in the SBRs for Run #2



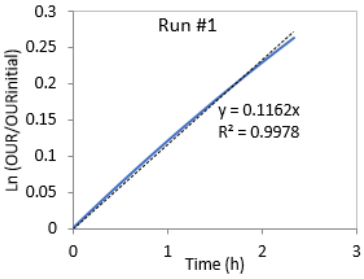
Decay coefficient was calculated based on the equation: $\ln OUR = -b_H t$

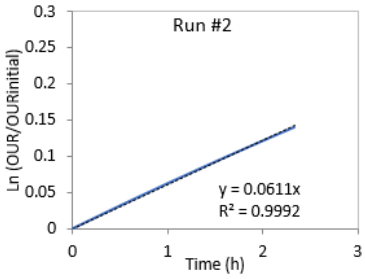


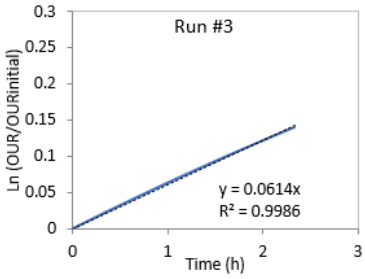
$b_H = 0.0184\ h^{-1} = 0.44\ d^{-1}$ $b_H = 0.014\ h^{-1} = 0.34\ d^{-1}$ $b_H = 0.0157\ h^{-1} = 0.38\ d^{-1}$

Maximum specific growth rate was determined based on equation: $\ln\frac{OUR}{OUR_{initial}} = (\mu_{max} - b_H)\,t$

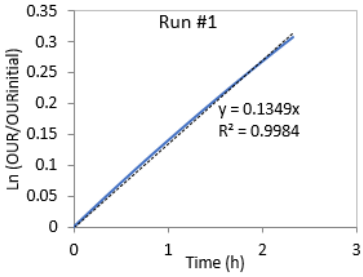
Raw wastewater (RWW)

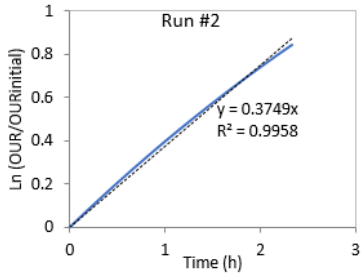


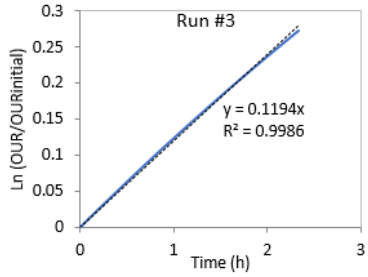




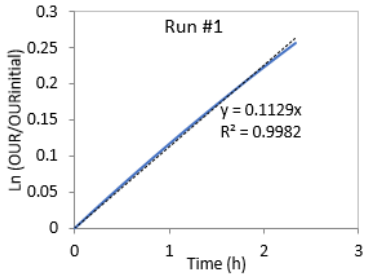
Primary clarifier effluent (PCE)

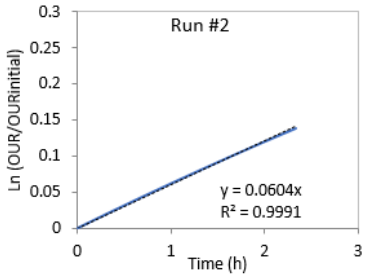


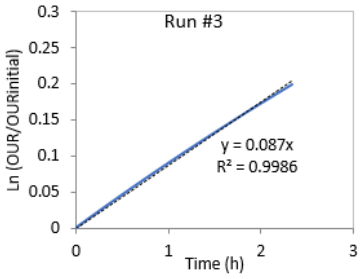




Rotating belt filter effluent (RBFE)

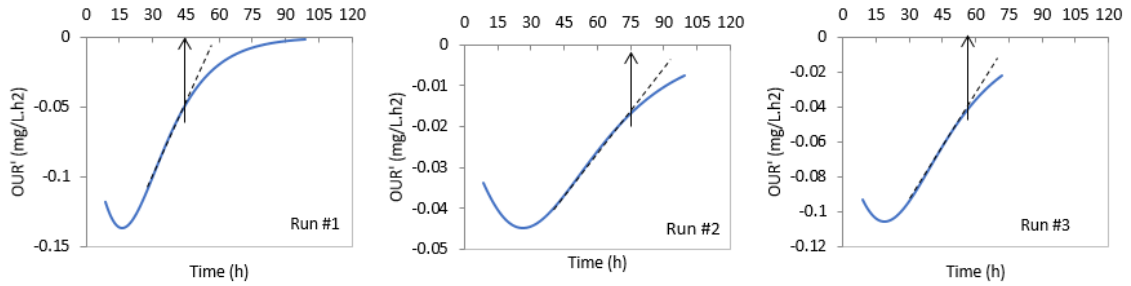




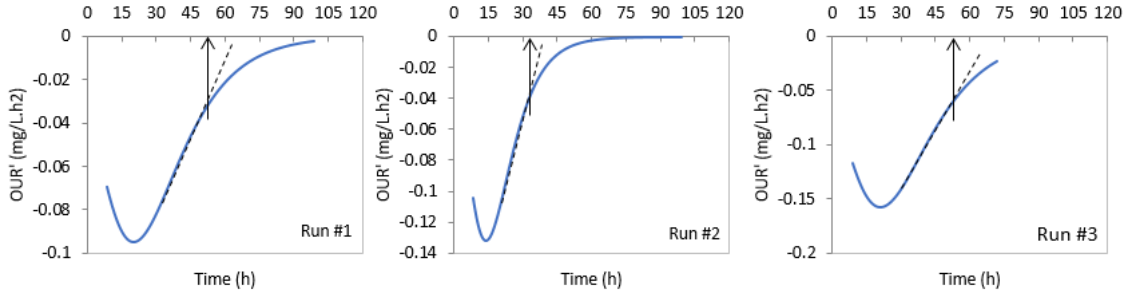


Readily biodegradable COD was determined based on equation: $S_S = \frac{\Delta O_2}{1-Y_H}$

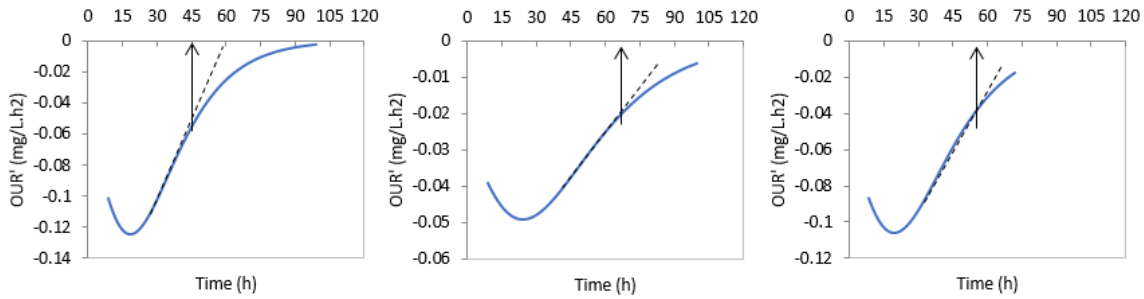
Raw wastewater (RWW)



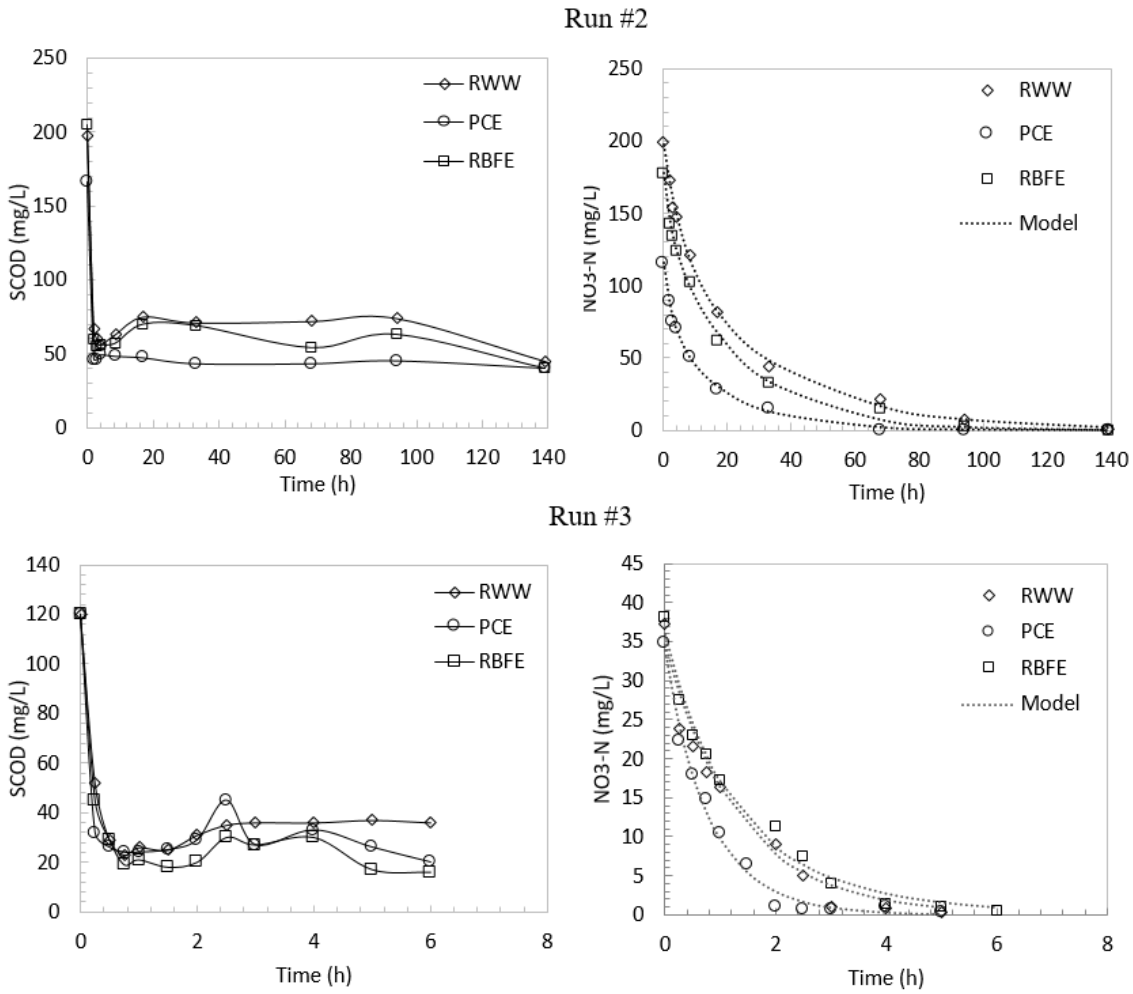
Primary clarifier effluent (PCE)



Rotating belt filter effluent (RBFE)



Batch NUR tests: SCOD and nitrate profile

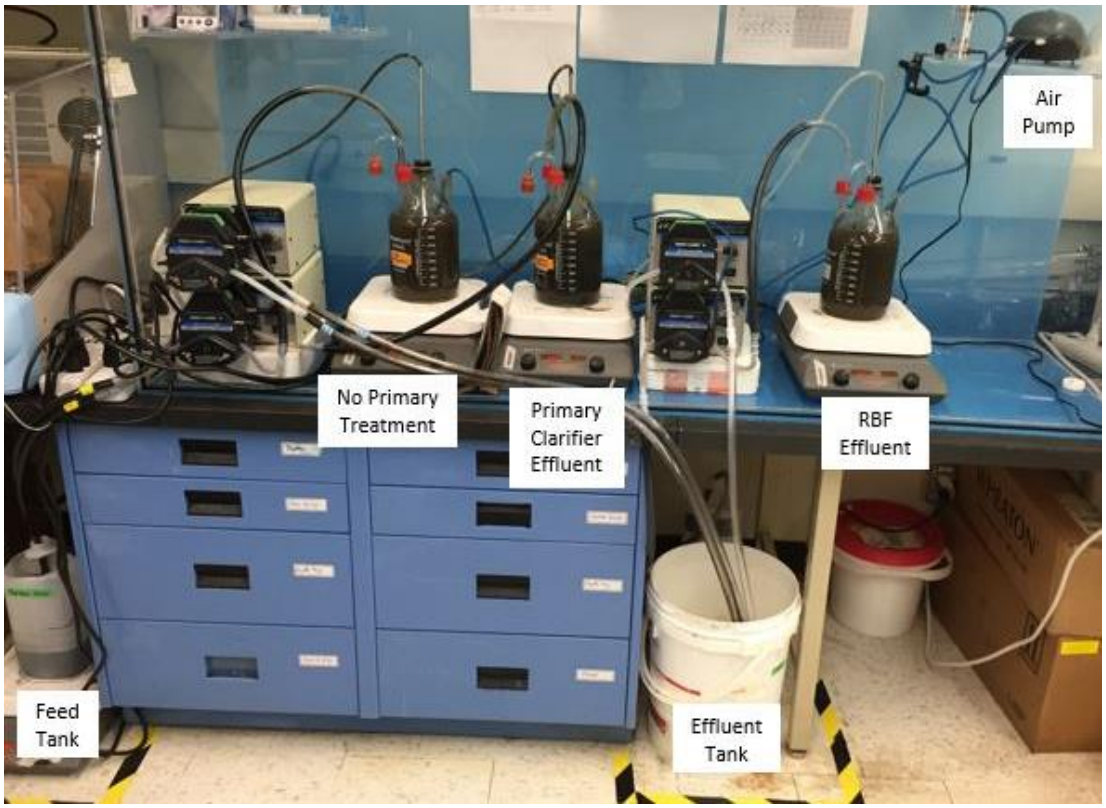


Appendix C. Supplementary material of Chapter 5

Table C4- 1. Input parameters and calculated concentrations based on the calibrated TSS/COD coefficients

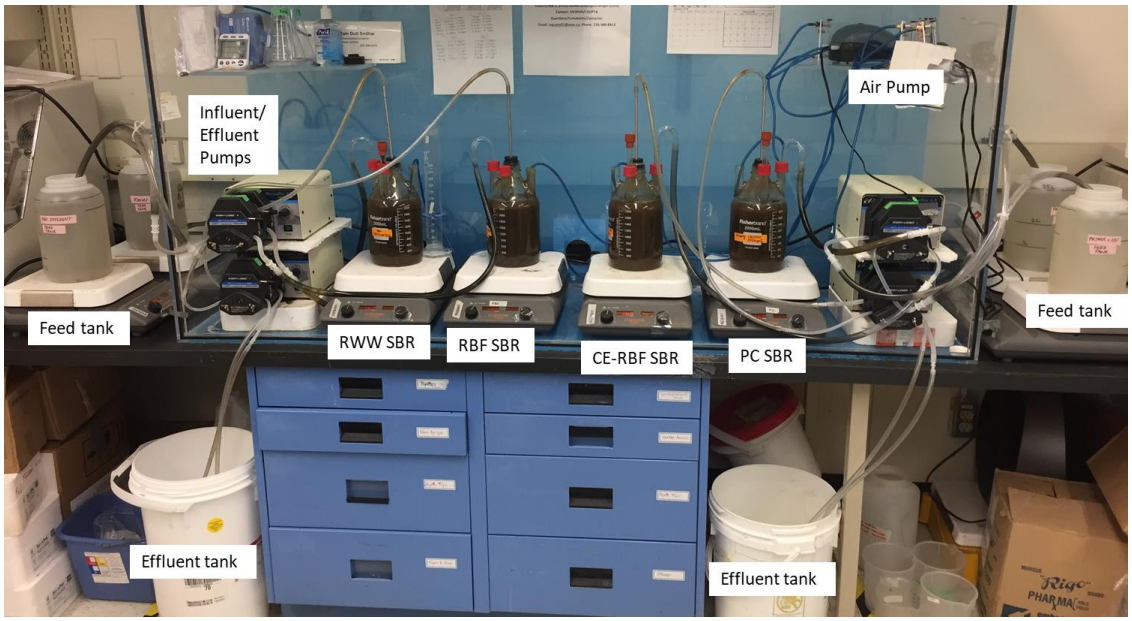
Parameters	Unit	Default	RWW SBR	PC SBR	RBF SBR
SBR input values					
TCOD	mg/L	n/a	750	490	610
TSS	mg/L	n/a	330	100	240
TKN	mg/L	n/a	56	44	51
Particulate COD/VSS	gCOD/gVSS	1.80	1.71	2.00	1.72
VSS/TSS	gVSS/gTSS	0.75	0.78	0.77	0.76
Inert fraction of sCOD, f_{Si}	-	0.20	0.12	0.11	0.12
Ammonium/TKN	-	0.63	0.70	0.70	0.68
Particulate COD/VSS (MLSS)	gCOD/gVSS	1.48	1.48	1.65	1.42
SBR kinetic parameters					
Maximum specific heterotrophic growth rate, μ_h	d^{-1}	3.2	6	6	6
Maximum specific autotrophic growth rate, μ_a	d^{-1}	0.9	0.9	1.2	0.9
Ammonia half-saturation coefficient, K_S	mg/L	0.7	1	1	1
Model calculated values					
VSS	mg/L	n/a	260	80	180
sCOD	mg/L	n/a	310	330	300
TN	mg/L	n/a	57	45	52
TP	mg/L	n/a	10	6	9
Soluble inert organic material, s_i	mg/L	n/a	37	37	36
Readily biodegradable substrate, s_s	mg/L	n/a	270	290	260
Particulate inert organic material, x_i	mg/L	n/a	79	32	55
Slowly biodegradable substrate, x_s	mg/L	n/a	360	130	250

SBR set-up

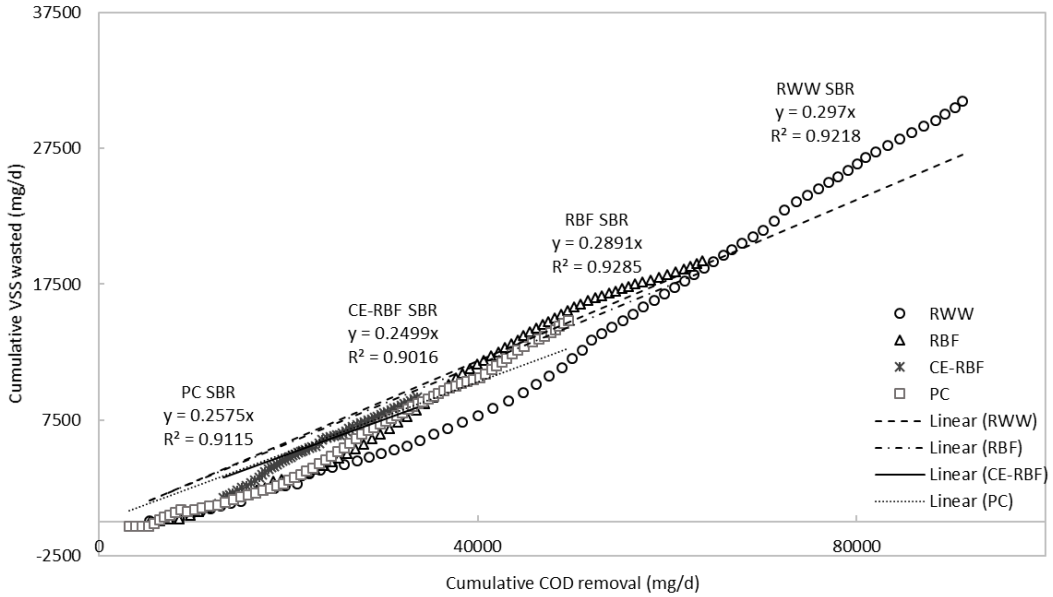

Air Pump
No Primary Treatment
Primary Clarifier Effluent
RBF Effluent
Feed Tank
Effluent Tank

Appendix D. Supplementary material of Chapter 6

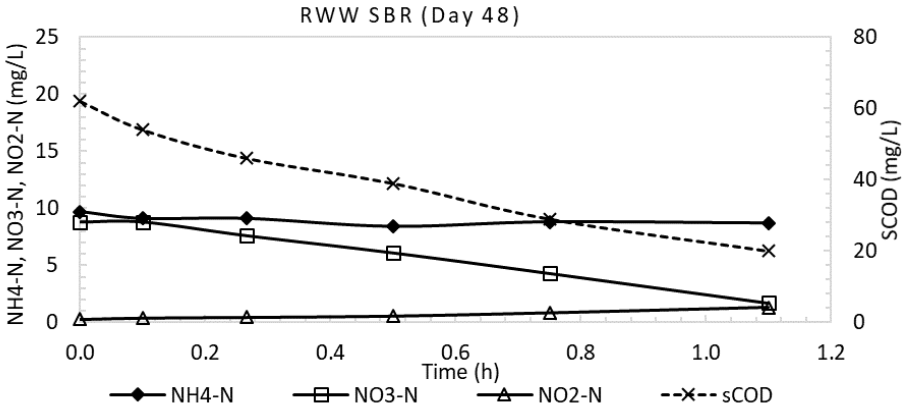
SBR set-up

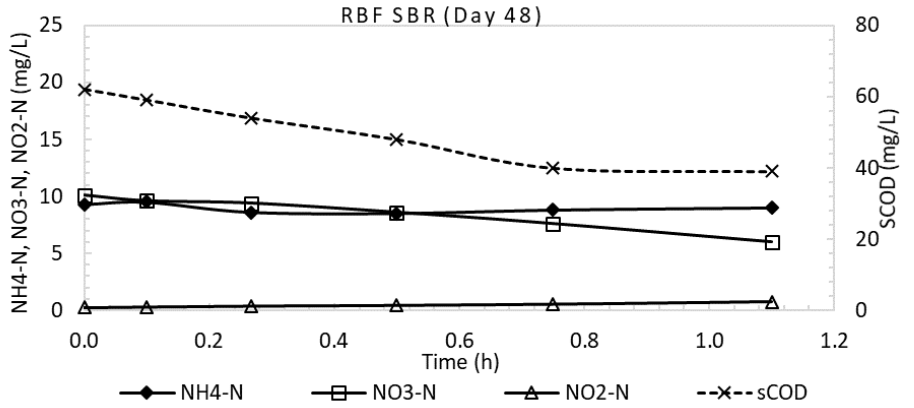


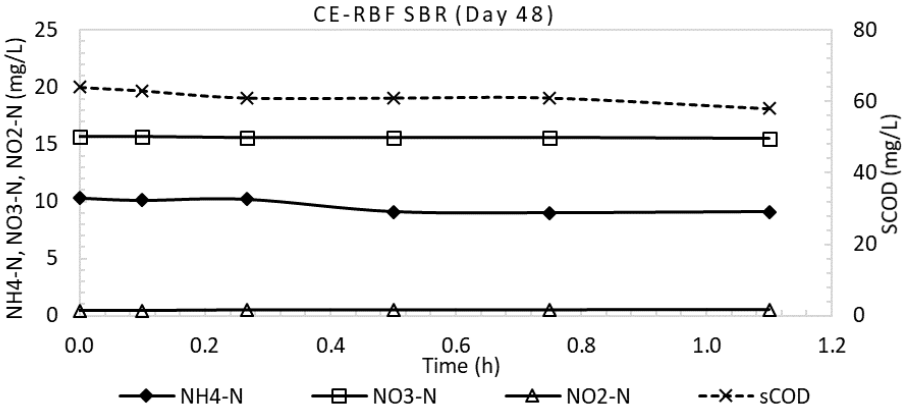
Cumulative biomass yield in the four SBRs

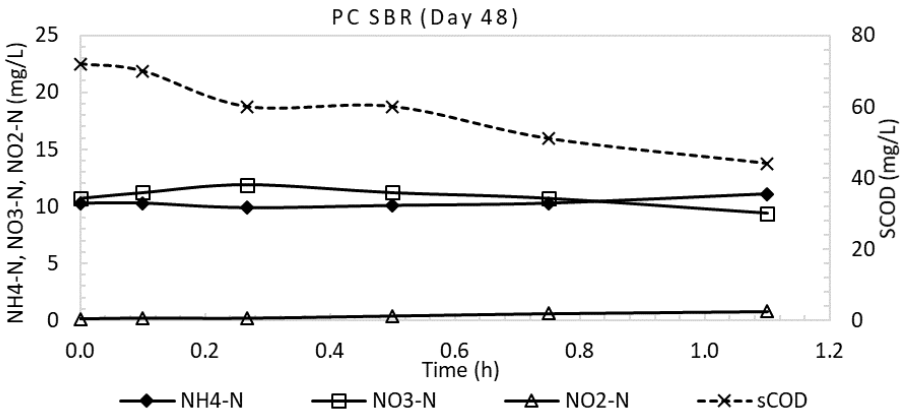


Cycle test on the four SBRs with sodium acetate feed to check for carbon limitation

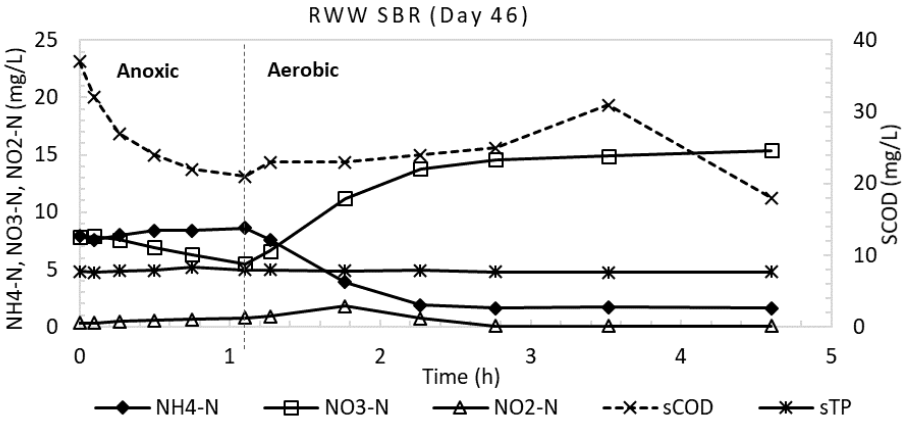


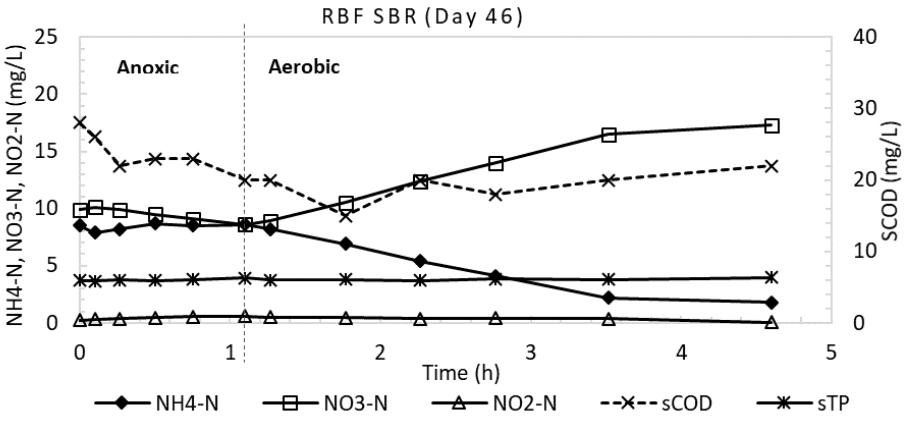


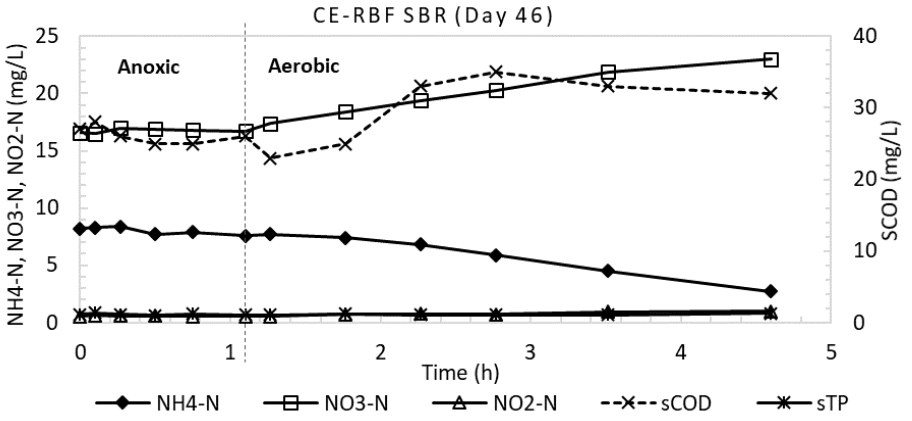


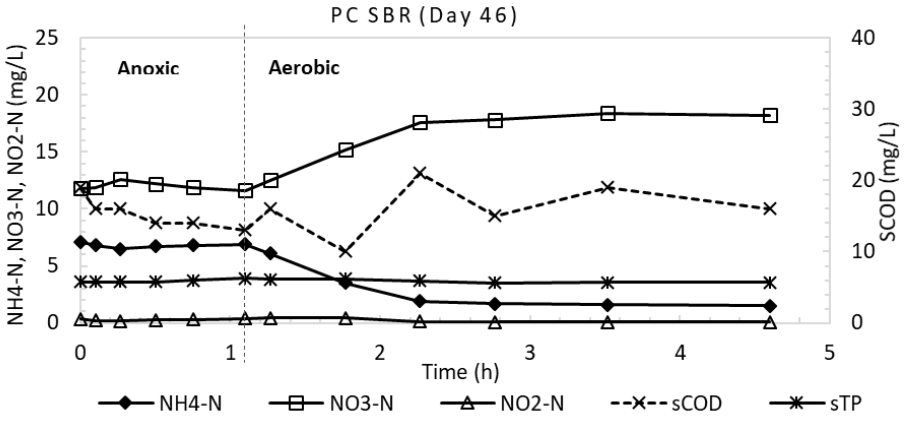


Cycle test on the four SBRs to determine nitrification and denitrification rates






CE-RBF SBR (Day 46)
Anoxic
Aerobic
NH4-N, NO3-N, NO2-N (mg/L)
SCOD (mg/L)
Time (h)
NH4-N
NO3-N
NO2-N
sCOD
sTP


PC SBR (Day 46)
Anoxic
Aerobic
NH4-N, NO3-N, NO2-N (mg/L)
SCOD (mg/L)
Time (h)
NH4-N
NO3-N
NO2-N
sCOD
sTP

Curriculum Vitae

Name:	Medhavi Gupta
Post-secondary Education and Degrees:	University of Windsor Windsor, Ontario, Canada 2008-2012 B.Sc. [Honours], Biochemistry and Biotechnology
	Western University London, Ontario, Canada 2012-2014 M.E.Sc. Chemical and Biochemical Engineering
	Western University London, Ontario, Canada 2014-2018 Ph.D. Chemical and Biochemical Engineering; Environmental and Green Engineering
Honours and Awards:	Western Graduate Research Scholarship 2012-2018
	Two-time 'Teaching Assistant of the Year' Award Tutorial Instructor: Wastewater Treatment Awarded by Chem Club, Western University 2014-2015, 2016-2017
	MITACS PhD Accelerate Scholarship with Trojan Technologies 2014-2016
	Second place award, Poster presentation Waterloo International Conference, Waterloo, ON, Canada 2014
Related Work Experience	Research Engineer Singapore**,** Trojan Technologies Aug-Nov 2014
	Research Associate Lystek International Inc. May 2013-Nov 2017
	Laboratory Coordinator Western University 2013-2018

Graduate Research Assistant
Western University
2012-2018

Graduate Teaching Assistant
Western University
2013-2018

Conferences and Publications:

Gupta, M., Sridhar, G., Wen, Y., Daynouri-Pancino, F., McLellan, A., Santoro, D., Nakhla, G. Evaluation of Chemically-Enhanced Microsieving on Nitrogen-Removal in WRRFs. 4th IWA Specialized International Conference, ecoSTP18, June 25-28, 2018, London, ON, Canada. (presenting author: Medhavi Gupta)

Gupta, M., Ho, D., Sarathy, S., Rosso, D., Batstone, D., Santoro, D., Nakhla, G. Microsieving wastewater using rotating belt filter technology to maximize internal carbon utilization for nitrogen removal. 53rd Central CAWQ Symposium, Feb 22, 2018, Toronto, ON, Canada. (presenting author: Medhavi Gupta)

Gupta, M., Ho, D., Sarathy, S., Rosso, D., Batstone, D., Santoro, D., Nakhla, G. (2017). The impact of sieving raw wastewater on nitrogen removal in wastewater resource recovery facilities. Revisions submitted to *Environmental Technology*.

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