

Impact of Powdered Activated Carbon on Anaerobic Membrane Bioreactors Treating Municipal Wastewater under Psychrophilic Conditions

by
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Author's Declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of my thesis, including any required final revisions, as accepted by my examiners. I understand that my thesis may be made electronically available to the public.

Abstract

The anaerobic membrane bioreactor (AnMBR) is an emerging technology, which has the potential for anaerobic digestion of wastewaters (WW) at high efficiency while generating biogas as a by-product (Harb et al., 2017). One of the main challenges associated with AnMBRs is reduced efficiency at low temperatures due to decreases in both bioreactor and membrane performance which makes using AnMBRs in North America impractical as WW temperature is typically in the range of 10 – 20°C (Environment Canada, 2017).

The addition of powdered activated carbon (PAC) could be a solution to the increased fouling and low chemical... (COD) removals that occur at low temperatures as it has abrasive and adsorptive properties. There are some studies that have observed the effect of PAC on AnMBRs resulting in increased quality of permeate but none tested varying concentrations of PAC at psychrotrophic temperatures using raw WW. Hence, this was investigated in this study.

Three-lab scale AnMBRS were set up in parallel and monitored for both bioreactor and membrane parameters over a two-phase experiment. Initially the membranes were operated at 10°C and 24°C to establish the performance under mesophilic and psychrophilic conditions without the presence of PAC. Then all three reactors were converted to psychrophilic conditions and three different concentrations of PAC were added to the reactors, 0.5, 1.0, and 2.0 g/L. To determine the effect of PAC on membrane performance, transmembrane pressure (TMP) and concentrations of organic sub-fractions were monitored. Bioreactor performance was evaluated by monitoring chemical oxygen demand (COD) and suspended solids (SS) in the waste activated sludge (WAS) feed and permeate.

Stage 1 of the research showed that membrane fouling increased at psychrotrophic temperatures as the cycle lengths decreased from 9 days at 24°C to 3 days at 10°C. Similarly, the bioreactor performance also decreased significantly with the temperature drop, resulting in lower permeate quality. Overall, the results were consistent with literature reports.

Stage 2 of the experimentation revealed that adding 1.0 g/L of PAC significantly lowered the level of SCOD in the WAS and TCOD in the permeate, resulting in a higher quality of permeate. Reduced performance with 2.0 g/L of PAC was attributed to the PAC acting as both an adsorbent (non biodegradable COD & colloidal COD) and as a scouring agent. At a low concentration (0.5 g/L) of PAC the adsorptive effect was not prominent. At the highest concentrations, PAC was adsorbing

and causing significant scouring as was evident through the increase in cycle lengths. However, it appears that the increased scouring reduced biofilm formation at the membrane and thus limited biodegradation that occurred on it thereby resulting in a lower quality of permeate and higher SCOD levels. Hence adding PAC at moderate concentrations was deemed to provide the best performance.

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Table of Contents

Author's Declaration	ii
Abstract	iii
Acknowledgements	v
Table of Contents	vi
List of Tables	viii
List of Figures	viii
List of Abbreviations	ix
Chapter 1 – Introduction	1
1.1 Background	1
1.2 Research Objectives	2
1.3 Thesis Outline	2
Chapter 2 – Literature Review	3
2.1 Introduction of AnMBRs	3
2.2 Effect of temperature on AnMBRs	4
2.2.1 Effect of Temperature on bioreactor performance	7
2.2.2 Effect of temperature on membrane performance.....	8
2.3 Effect of Activated Carbon in MBRs	9
Chapter 3 – Methodology and Materials	16
3.1 Test Plan	16
3.1.1 Outline	16
3.1.2 Experimental Configuration.....	18
3.2 Operation of AnMBRs	20
3.2.1 Influent for Anaerobic digestion.....	20
3.2.2 Maintenance procedures	21
3.2.3 Membrane Cleaning Procedure	21
3.2.4 Sampling Procedure.....	22
3.2.5 Sample Analysis.....	23
Chapter 4 – Results and Discussion	25
4.1: Influent Characteristics	25
4.2 Phase 1 Results	29
4.2.1 Validation	29
4.2.2 Analysis and Discussion.....	30
4.2.3 Conclusions of Phase 1	42
4.3 Phase 2 of Experimental Analysis	43
4.3.1 Validate Phase 2 Data.....	43
4.3.2 Analysis and Discussion.....	44
4.3.3 Conclusion of Phase 2	57
5.0 Conclusions and Recommendations	57

5.1 Conclusions	57
5.2 Recommendations.....	60
Reference	61
Appendixes.....	65

List of Tables

Table 2. 1: Performances obtained in prior AnMBR studies for DWW treatment.	6
Table 2. 2: Summary MBR studies using PAC to treat Wastewater	12
Table 3. 1: Sample Schedule.....	22
Table 4. 1: TCOD Averages for Phase 2.....	45
Table 4. 2:SCOD in WAS for Phase 2	46
Table 4. 3: Average permeate TCOD for phase 2.....	49
Table 4. 4: Average cycle length for Phase 2	56

List of Figures

Figure 4. 1: TCOD Concentrations in the influent during the research period	25
Figure 4. 2: SCOD Concentrations in the influent during the research period.....	26
Figure 4. 3: Concentration of TSS in the influent during the research period.....	27
Figure 4. 4: Concentration of VSS in the influent during the research period	28
Figure 4. 5: TCOD concentration WAS for all three reactors.....	30
Figure 4. 6: Cumulative TCOD in WAS in phase 1 for all three reactors	32
Figure 4. 7: The SCOD concentration of all three reactors in Phase 1	33
Figure 4. 8: Suspended Solids for All three reactors in phase 1.....	34
Figure 4. 9: COD concentrations of the three reactors in phase 1.....	36
Figure 4. 10: The Average and standard deviation of permeate quality in all three reactors	36
Figure 4. 11: COD distribution in the three reactors during phase 1	38
Figure 4. 12: Humics concentration in phase 1.....	39
Figure 4. 13: Biopolymers concentrations for phase 1	40
Figure 4. 14: Concentration of Building Blocks over phase 1	41
Figure 4. 15: Frequency distribution of cycle lengths of each reactor	42
Figure 4.16: TCOD in the WAS for phase 2.....	45
Figure 4.17: SCOD in WAS in all reactors for phase 2	45
Figure 4.18: TSS and VSS in reactors during phase 2	48
Figure 4.19: COD in permeate samples for reactors I phase 2.....	49
Figure 4.20: COD distribution in all reactors in phase 2	51
Figure 4.21: Humics concentrations in the WAS during Phase 2	52
Figure 4.22: Biopolymer concentration in Phase 2	53
Figure 4. 23: Building Blocks concentration in all reactors in phase 2	54
Figure 4. 24: Frequency plot for cycle length	55
Figure 4. 25: TMP for ABC for a cycle.....	56

List of Abbreviations

AnMBRs: Anaerobic membrane bioreactor
ANOVA: Analysis of Variance
bCOD: Biodegradable chemical oxygen demand
BP: Back-pulse
BB: Building blocks
BP: Bio-polymers
COD: Chemical oxygen demand
CF: Cleaning frequency
DOC: Dissolved organic carbon
EPS: Extracellular polymeric substances
HRT: Hydraulic retention time
LC-OCD: Liquid chromatography- Organic carbon detection
LMW: Low molecular weight
LMWA: Low molecular weight acids
LMWN: Low molecular weight neutrals
MBR: Membrane bioreactor
MC: Maintenance clean
MWW: Municipal waste water
nbCOD: Non-biodegradable chemical oxygen demand
nbpCOD: Non-biodegradable particulate chemical oxygen demand
nbsCOD: Non-biodegradable soluble chemical oxygen demand
RC: Recovery clean
rbCOD: Readily biodegradable chemical oxygen demand
sbCOD: Slowly biodegradable chemical oxygen demand
SRT: Solids residence time
SCOD: Soluble chemical oxygen demand
TCOD: Total chemical oxygen demand
TMP: Transmembrane pressure
TOC: Total organic carbon
TSS: Total suspended solids
VSS: Volatile suspended solids
UF: Ultrafiltration
WW: Wastewater
WAS: Waste activated sludge

Chapter 1 – Introduction

1.1 Background

The anaerobic membrane bioreactor (AnMBR) is an emerging technology, which has the potential for anaerobic digestion of wastewaters (WW) at high efficiency while generating biogas as a by-product (Harb et al., 2017). AnMBRs combines anaerobic digestion with membrane filtration. Anaerobic digestion consists of a series of biological processes in which microorganisms break down biodegradable organic material in the absence of oxygen leading to the formation of digestate and biogas which mainly consist of methane and carbon dioxide (Horan et al., 2018). The membrane is submerged in the anaerobic reactor and is used to generate a permeate that is separated from the MLSS allowing for a separation of the hydraulic and solids residence times.

A challenge associated with AnMBRs is reduced efficiency at low temperatures because temperature affects bioreactor performance. Thus using AnMBRs is a challenging option in North America as the WW temperature is typically in the range of 10 – 20°C (Environment Canada, (2017)). The literature (Wen et al., 1999; Chu et al., 2005; Ho et al., 2010) has demonstrated significant reductions in COD removal with over a 5 – 10°C change in temperatures and reported COD percentage removals were lower than 90% when the temperature decreased below 20°C . Wen et al. (1999) concluded that COD removal rates are negatively influenced by reductions in temperature.

In addition to reduced bioreactor performance at low temperatures it was found that membrane fouling also increased, as temperature effects the viscosity of the MLSS and the solubility of various compounds and gases which in turn can effect fouling rates of membranes. Martinez-Sosa et al. (2011) found that the operational temperature of an AnMBR is related to the observed membrane fouling. Increased membrane fouling combined with low bioreactor performance at low temperatures resulted in poor efficiencies.

A potential solution to the increased fouling and low COD removals that at occurs at low temperatures could be the addition of PAC as it has abrasive and absorptive properties. Ying et al. (2006) investigated the effect of PAC dosage on retarding membrane fouling in an aerobic MBR. A decrease in the formation of EPS was observed when a PAC dosage of 0.75mg/L was added to the

MBRs, which in turn resulted in higher permeate quality with lower levels of COD. Further, Remy et al. (2006) evaluated low dose PAC addition at high SRTs to reduce fouling in membrane bioreactors. It was concluded that PAC significantly lowered the TMP across the membrane, thus increasing the operation by minimizing cleaning frequencies. Several other studies (Ying and Ping, 2006, Munz et al., 2007, Liu et al., 2007, Lesage et al., 2008, Remy et al., 2010) suggest that PAC addition improved COD removal, although in some cases the effect was marginal, especially for WWs with low concentrations of biodegradable compounds. Overall, the literature suggests that PAC could be used to increase the efficiency at lower temperatures.

1.2 Research Objectives

The purpose of this research project was to explore the effect that powdered activated carbon (PAC) has on the performance of AnMBRs in psychrotrophic conditions. The following specific research questions were addressed.

- Does PAC addition affect the extent of anaerobic digestion in AnMBRs at low temperature?
- Does PAC addition affect membrane performance under these conditions?
- Is there a relationship between PAC dosage and these responses?

1.3 Thesis Outline

This thesis consist of five Chapters. Chapter 2 consists of a literature review, which is an overview about of the published material relevant to this project. Based on the findings the experiment was determined in order to fill in the gap. Chapter 3 outlines the research methodology that was undertaken for the study. Chapter 4 presents a detailed analysis of the results. Chapter 5 presents conclusions and recommendations drawn from the entire project.

Chapter 2 – Literature Review

The literature review presented in this Chapter provides a detailed background of the significant research conducted prior to this project. It begins with a general introduction of AnMBRs. Section 2.2 presents an analysis of the impact of temperature on both the membrane and bioreactor responses. Section 2.3 introduces the properties and potential benefits of using PAC to reduce fouling. In this section, various studies on MBRs using PAC to mitigate fouling were evaluated in terms of both membrane and bioreactor performances collectively.

2.1 Introduction of AnMBRs

Anaerobic digestion consists of a series of biological processes in which microorganisms break down biodegradable material in the absence of oxygen. One of the products is biogas, which can be combusted to generate electricity and heat, or can be processed into renewable natural gas and transportation fuels (Metcalf and Eddy, 1979). Hydrolysis is often the rate-limiting step in anaerobic digestion (Bougrier et al., 2006). In addition, due to the slow growth rates of methanogens, conventional anaerobic digesters need to be operated at long residence times (Dagnew, 2010). One major drawback of operating digesters at long residence times is that the volumes of the reactors are large, which in turn increases the costs associated with the construction and maintenance of the digester. Collectively, these factors act to increase the cost of conventional digestion.

One way to enhance anaerobic digestion is to incorporate a membrane into the design of the digester (Pickel, 2010). With an anaerobic membrane bioreactor (AnMBR), the solids retention time (SRT) can be decoupled from the hydraulic residence time (HRT), thus resulting in the ability to treat at lower HRTs thereby occupying less space in the WWTP. The membrane is able to retain the biomass and microorganisms in the digester resulting in a waste sludge with high solids concentration. Moreover, the permeate effluent is free of solids due to the membrane operation (Lew et al., 2009). Due to all these advantages, AnMBRs are gaining popularity in the wastewater industry.

A challenge associated with AnMBRs is low efficiency at low temperatures because temperature affects both bioreactor and membrane performance. Thus AnMBRs have not been employed in North America for wastewater treatment as wastewater temperatures are typically in

the range of 10 – 20°C (Environment Canada, 2017). Section 2.2.1 analyzes prior studies of the effect temperature on bioreactor performance, as demonstrated by parameters such as COD in the WAS and effluent and removal efficiencies. Similarly, Section 2.2.2 investigates the effect of temperature on the membrane performance.

One potential solution to increased fouling is the utilization of activated carbon as suggested by Hu and Stuckey (2007), who demonstrated the use of both PAC and granular activated carbon (GAC) as an abrasive and an absorbent. Thus, these aspects have been investigated in the later sections.

2.2 Effect of temperature on AnMBRs

A summary of prior research into the treatment of domestic wastewater (DWW) using AnMBR's is presented in Table 2.1. The selection of research cited, while not exhaustive, is representative of the work done in this area based on their collective range of operating temperatures. The studies intended to optimize AnMBRs (measured as %COD removal) using variables such as HRT, SRT, and membrane configuration, and were selected in order to represent results across a wide range of operating temperatures below 37°C. Although temperatures of 24°C or above are not within the test range for the present study, despite being the conventional operating temperature for anaerobic digesters (Metcalf and Eddy, 2003), the effect of temperature on AnMBR's in general was of interest for the purpose of identifying trends and consequences of operating at a lower temperature. The selected studies used performance indicators other than % COD removal but this one parameter was consistent throughout, facilitating a salient comparison.

While the literature shows that bioreactors operate more efficiently at mesophilic temperatures, the impact of temperature combined with other seasonal variations has not been explored thoroughly. This is, in part, due to the use of simulated WW in the studies cited in Table 2.1, with the exceptions of Baek & Pagilla (2006) and Wen et al. (1999). Simulated WW studies typically do not account for the effect of seasonal variations on the system. These variations include the rate of temperature fluctuation, organic composition, and organic sub-fractions (humics, biopolymers, LMW neutrals, and building blocks). The use of raw WW allows the combined effects of these variables to be more clearly observed and accounted for.

Table 2. 1: Performances obtained in prior AnMBR studies for DWW treatment

Study	Average influent strength (mg/L TCOD)	Type of wastewater	Bioreactor configuration	Membrane information	Fouling control	SRT (d)	HRT (h)	Average effluent (mg/L)	% COD removal	Temp (°C)
Wen et al. (1999)	100-2600	Raw	UASB with submerged membrane	0.03 µm polyethylene submerged hollow fiber	Periodic cleaning with 5% NaOCl	∞	4 to 6	12 - 19	97	12 - 25
Chu et al. (2005)	383-849	Synthetic	EGSB with submerged membrane	0.1 µm polyethylene submerged hollow fiber	Back flushing and relaxation; periodic cleaning with 0.03% NaOCl	∞	3.5-5.7	93	78	11
								85	86	15
								93	96	25
								87	92	20
Hu and Stuckey (2006)	460	Synthetic	Submerged AnMBR	0.4 µm Submerged hollow fiber, 0.4 µm polyethylene chloride submerged sheet	Biogas sparging	∞	3	44 - 43	90 - 91	35
							6	40 - 40	91 - 91	
							12	38 - 32	92 - 93	
							24	29 - 32	94 - 93	
							48	23 - 25	95 - 96	
Baek and Pagilla (2006)	84 [SCOD]	Raw	Completely mixed anaerobic bioreactor	0.1 µm PVDF external tubular	Cross-flow; weekly cleaning with 0.1% w/w NaOH and disinfectant	∞	12	24	68	32
							16	24	68	
							24	37	55	
							48	25	58	
Saddoud et al. (2007)	685	Raw	Jet flow anaerobic reactor	100 kDa external	Cross-flow	∞	15 - 60	87	88	37
Ho and Sung (2009)	500	Synthetic	Completely mixed anaerobic reactor	1 µm PTFE external tubular	Cross-flow; periodic cleaning with NaOCl	90-360	6 to 12	<40	>92	25
Ho and Sung (2010)	500	Synthetic	Completely mixed anaerobic reactor	1 µm PTFE external tubular	Periodic backflushing	∞	9	25	95	25
								75	85	15

Study	Average influent strength (mg/L TCOD)	Type of wastewater	Bioreactor configuration	Membrane information	Fouling control	SRT (d)	HRT (h)	Average effluent (mg/L)	% COD removal	Temp (°C)
Lew et al. (2009)	540	Synthetic	Completely mixed anaerobic reactor	0.2 µm external hollow fiber	Periodic back flushing; chemical cleaning with 0.1 M NaOH, 1% H ₂ O ₂ , and 1% HCl	∞	4.5 - 12	65	88	25
Gao et al. (2010)	500	Synthetic	Upflow anaerobic reactor	100 kDa external coated PVDF and 30 kDa external polyetherimide	Cross-flow	50	24	<20	>96	30
Huang et al. (2011)	550	Synthetic	Completely mixed anaerobic reactor	0.45 µm PESf flat sheet	Biogas sparging	30	8 to 12	<17	>97	25-30
						60				
						∞				
Salazar-Pelaez et al. (2011)	350	Synthetic	UASB with external membrane	100 kDa external PVDFd tubular	Cross-flow; NaOCl cleaning every 6 h	∞	4 to 12	70	80	-
Kim et al. (2011)	513	Synthetic	Two-stage fluidized bed/membrane bioreactor	0.1 µm PVDF hollow fiber	GAC fluidization; periodic back flushing and/or NaOCl/NaOH cleaning	∞	4.2 - 5.9	7	99	35
Smith et al. (2011)	440	Synthetic	Submerged AnMBR	0.2 µm PES flat sheet	Biogas sparging and back flushing	300	16	36	81	3
									90	6
									90	9
									90	12
									90	15

2.2.1 Effect of Temperature on bioreactor performance

The literature review examined studies spanning operating temperatures from 3°C (Smith et al., 2011) to 37°C (Saddoud et al., 2007). As Table 2.1 shows, most prior studies of AnMBRs have involved operation at mesophilic temperatures. Of all the studies summarized therein, only three studies were operated at psychrophilic temperatures. Chu et al. (2005) and Ho et al (2010) operated AnMBRs at 15°C and achieved COD removals of 76% and 85%, respectively. Smith et al. (2011) conducted a series of experiments at temperatures between 3°C to 15°C and reported COD removals of 90% for all temperatures above 3°C, which was considerably higher than the other studies. However, all the cited studies that operated at psychrophilic temperatures used simulated WW. Due to the aforementioned inability of such experiments to account for the seasonal variations in raw DWW, they leave a gap in the available research.

Chu et al. (2005) and Ho et al. (2010) compared operations at 15°C to warmer temperatures of 20°C and 25°C thus a relationship could be outlined with regards to temperature. Chu et al. (2005) used an EGSB submerged membrane with a 0.1 µm polyethylene hollow fiber with regular back flushing and relaxations and periodically cleaning with 0.03% NaOCl. They tested the membrane at 25°C, 20°C, 15°C and 11°C and observed an overall 20% reduction in percentage COD removal efficiency with values of 96%, 92%, 86% and 76%, respectively. Similarly, Ho et al (2010) used a completely mixed anaerobic reactor with a 1 µm polytetrafluoroethylene (PTFE) external tubular membrane with periodic back flushing and achieved lower % COD removals at 15°C and 25°C with removal of 85% and 95% respectively. The difference in removal efficiencies between the studies may have been due to the operational factors; for instance, the frequency of cleaning and backwashing. Both studies noted that there was a reduction in % COD removals as the temperatures decreased but a relationship was not concluded. Ho et al. attributed the decrease to suppressed methanogenic activity at lower temperatures.

Wen et al (1999) reported high COD removal efficiencies at temperatures ranging from 12°C to 25°C. A dependency on temperature was observed, as COD percentage removals decreased to below 70% at temperatures below 15°C which will be elaborated upon later. Chu et al., (2005) similarly observed a sharp decline to 78% COD removal when the temperature was reduced to 11°C. The two studies used similar HRT values (3.5–5.7 hours in Chu et al. and 4-6 hours in Wen et al.), which may be the reason for similarity between the data but they used different membrane

configurations with different pore sizes, which may account for the slight variance hence the COD removal efficiencies were not identical. In conclusion, reducing temperature has been found to negatively affect COD removal rates and permeate quality, but there is limited detailed research at psychrophilic temperatures while controlling for other factors.

Smith et al. (2011) conducted a series of experiments at temperatures between 3°C to 15°C and reported a COD removal efficiency of 90% for all temperatures above 3°C. This was in contrast with all other studies at psychrophilic temperatures that observed a significant drop in COD removal after each incremental temperature decrease of 5°C – 10°C. Typically, removal efficiencies have been found to decrease below 90% once the temperature is decreased to 20°C. Not only did Smith et al. achieve 90% removal at 15°C, this removal efficiency was maintained until the temperature reached 6°C. The difference in results was explained by the formation of a biofilm on the membrane. A significant amount of biofilm was allowed to form on the membrane surfaces and the contribution of the membrane biofilms to biological treatment increased as temperature decreased. This in turn decreased the suspended biomass. High dissolved methane oversaturation occurred due to an increase in methanogenesis in the biofilm at lower temperatures. This resulted in low concentrations of COD in the permeate. However, allowing the biofilm to develop increased the pressure required for membrane filtration and is typically not practical in the field.

2.2.2 Effect of temperature on membrane performance

Temperature affects not only the rate of the biodegradation process but also the viscosity of the MLSS and the solubility of various compounds and gases which in turn can affect fouling rates of membranes. Martinez-Sosa et al. (2011) found that the operational temperature of an AnMBR influenced membrane fouling. The researchers operated an AnMBR system at a flux of 7 L/m² h at 35°C, and at 20°C and observed an increase in the fouling rate at 20°C. This was attributed to TSS and soluble COD accumulation and a higher viscosity in the bioreactor. The fouling rate was reported as 0.14 mbar/d at 35°C, whereas it was 2.61 mbar/d at 20°C. The decrease in temperature and increase in solids content were proposed as possible explanations for the viscosity increase and the increase in membrane fouling.

Membrane fouling is a major issue that occurs in both mesophilic temperatures and psychrophilic temperatures. This can occur due to internal and cake fouling. Chu et al (2005)

evaluated this and found that cake layer resistance was the major resistance. This finding was similar to that reported by Choo et al (1996) who investigated the fouling mechanisms by EPS. Although not many studies investigated the cause of fouling, several studies (Wen et al., 1999; Simit et al., 2011; Chu et al., 2005) have investigated methods to minimize cake fouling.

Wen et al. (1999) identified that the transmembrane pressure exerted on the membrane will increase with operation time due to membrane fouling at low temperatures and this can be mitigated by operating the system operating intermittently. Simit et al. (2011) addressed fouling by operating at subcritical flux. Similar to Wen et al. (1999), Chu et al. (2005) also incorporated operation of the effluent pump intermittently. Chu et al. (2005) attempted to reduce membrane fouling by increasing the immersion time of the membrane during cleaning, back washing twice a day for six minutes. Both methods successfully reduced cake formation on the membrane. Overall, cake fouling has been found to be a major concern and all methods to reduce it work, but they have to be further investigated to determine what is practical in reality based on economics.

In all cited studies, membrane fouling was found to increase as temperature decreased. The biofilm that develops on membranes can play a role in membrane fouling but it can also marginally improve SCOD removal rates and thus the final quality of the permeate. Several studies have illustrated a significant difference in SCOD concentrations between the permeate versus the mixed liquor inside the reactor (Chu et al., 2005; Hu and Stuckey, 2006; Ho and Sung, 2009, 2010; Baek et al., 2010; Smith et al., 2011). In addition, Ho and Sung (2010) observed an increase in SCOD removal across the membrane surface with decreasing temperatures, which could be due to the accumulation of biofilm. Other researchers (Ng et al., 2000; Baek and Pagilla, 2006; Ho and Sung, 2009) reported similar findings. The consensus over the studies is that the biofilm accumulation increases as temperature decreases and it can reduce the SCOD concentration in the permeate.

2.3 Effect of Activated Carbon in MBRs

The use of AC in MBR's, initially suggested by Hu and Stuckey (2007), was reviewed to provide insight into its possible effect as an abrasive in AnMBR's as a solution to fouling. Khan et al (2011) observed lower biofouling tendencies in hybrid MBR's using powdered activated carbon (PAC) when compared with MBR's using cationic polymer. Both these studies indicated that the effects of PAC are beneficial. In the literature review, reports on prior studies that used PAC in

AnMBRs for raw WW at low temperatures were not found so two recent studies that used PAC in MBRs (Ying et al (2016), and Remy et al. (2016) were evaluated in detail to obtain insights that have been derived in similar systems. Upon completion of this review, prior literature that studied the use of PAC in waste streams and MBRs at mesophilic temperatures was reviewed in Table 2.2.

The two main options for AC that can be used in this context are GAC and PAC. Hu and Stuckey (2007) compared three lab-scale reactors to investigate them (one was a control). The COD removal rate was greater than 90% in all three reactors but the average COD removal was higher in the PAC-assisted MBR than in the GAC-assisted MBR, which was not significantly better than that of the control MBR. Thus, moving forward only the use of PAC was evaluated.

Ying et al. (2016) and Remy et al. (2016) evaluated the impact of PAC when aerobically treating DWW at mesophilic temperatures. Ying et al. (2016) used three different concentrations of PAC of 0, 0.75 and 1 g/L. A slight decrease in COD and ammonia nitrogen in the effluent was observed with the addition of PAC. However, the reactor with a PAC addition of 0.75g/L had the lowest EPS build up, and hence the researchers concluded that PAC at a precise dosage was effective at reducing irreversible fouling but had no impact on the effluent. However, there are several things that could have been considered, for instance, the relationship between PAC concentration and fouling or effluent quality might not have been linear. Notably, they did not report the TMP, which would be expected to increase with EPS content. An investigation of the response of TMP as a function of EPS content and PAC dosage would have provided an improved assessment of the effect of PAC on overall membrane and bioreactor performance. This was done by Remy et al. (2016) who observed a significant reduction in TMP in the reactor with 1g/L PAC compared to a reactor without any PAC. The reactor without PAC began fouling at the 80th hour and completely fouled by the 160th hour whereas the reactor with the PAC still had not begun fouling at the 160th hour. Concurrently, it was found that significant fouling occurred without the addition of PAC. Moreover, there was a decrease of 10% in the critical flux level when PAC was employed. This was also validated by the protein and polysaccharides concentrations in the permeate as seen by Ying et al. (2016). Nevertheless, both the of studies support the hypothesis that using PAC should have an overall positive effect on MBR performance, since it prevents fouling without having a negative effect on permeate quality. Another point that can be taken from Remy et al. (2016) is that low dosage and high retention time make it feasible and cost effective to apply the technology to WW treatment plants if the results can be replicated in colder temperature and anaerobic conditions.

Table 2. 2: Summary MBR studies using PAC to treat wastewater

Scale	Type of wastewater	Volume (L)	MLSS (gL ⁻¹)	SRT (d)	HRT (h)	Dosage (gL ⁻¹)	Initial COD (average) (mg L ⁻¹)	Final COD (average) (mg L ⁻¹)	COD reduction (%)	References
Lab/A	Secondary effluent	37.5	4.5	-	4	0.75	10.7 ± 1.32 (TOC)	8.98 ± 0.62 (no PAC)	16.1 ± 4.6 (no PAC)	Lin et al. (2011)
								4.0 ± 0.84 (with PAC)	62.7 ± 7.7 (with PAC)	
Pilot/A	Tannery wastewater	0.52 m ³	12-18 (TSS)	32-95	50-100	0-3	4051	-	Low (but not negligible improvement) with PAC	Munz et al. (2007)
Pilot/A	Municipal wastewater	85	9.6 (no PAC)	25-50	10	0.5 (in terms of sludge)	300	33 (no PAC)	-	Remy et al. (2010)
			10.1 (with PAC)					30 (with PAC)		
Lab/A	Synthetic wastewater	2	12 ± 1 (no PAC)	30	4	0-5	370 ± 10 (TOC)	15 (TOC) (no PAC)	-	Ng et al. (2006)
			17 ± 1 (with PAC)					5 (TOC) (with PAC)		
Lab/A	Pharmaceutical wastewater	35	-	-	~ 6	0.25	575-3201	-	89.27 (no PAC) 89.79 (with PAC)	Liu et al. (2007)
Lab/A	Domestic wastewater	24	-	30	-	0-1.5	271.44-575.24	-	29.15-47.26 (no PAC) 24.36-47.52 (with PAC)	Ying and Ping (2006)
Lab/A	Low-strength synthetic wastewater	2 (effective)	-	-	2	0-75	5-6.5 (DOC)	4.64 (DOC) (no PAC)	80% (with PAC)	Ma et al. (2012)
								1.16 (DOC) (with PAC)		
								32 (with PAC)		

Scale	Type of wastewater	Volume (L)	MLSS (g L ⁻¹)	SRT (d)	HRT (h)	Dosage (g L ⁻¹)	Initial COD (average) (mg L ⁻¹)	Final COD (average) (mg L ⁻¹)	COD reduction (%)	References
Lab/A	Synthetic wastewater	-	9	20	24	0.2 g d ⁻¹	-	-	94 ± 2 (no PAC) 96 ± 2 (with PAC)	Lesage et al. (2008)
Lab/A	Sewage-contaminated surface water	2 (effective)	-	20	0.5	8 (mg L ⁻¹)	4.13 ± 0.37	2.66 ± 0.29 (no PAC) 1.66 ± 0.21 (with PAC)	35.3 ± 7.6 (no PAC) 59.5 ± 6.8 (with PAC)	Tian et al. (2008)
Lab/An	High salinity synthetic sewage	3 (effective)	-	250	8	1.7	145 ± 10 (DOC)	-	93% (no PAC) 98% (with PAC)	Vyrides and Stuckey (2009)
Lab/An	Municipal solid wastes leachate	10	4.4	30	1.5	2	5000	2380 (SCOD) (no PAC) 1550 (with PAC)	-	Trzcinski and Stuckey (2010)
Lab/An	Low-strength synthetic wastewater	3	2.6 ± 0.13 (no PAC) 3.7 ± 0.19 (with PAC)	150	6	1.7	450 ± 20	18 ± 11 (no PAC) 18 ± 9 (with PAC)	-	Aquino et al. (2006)
Lab/A	Biologically treated swine wastewater	22.1	-	-	2.5	1-10	217	172 (no PAC)	88.7 (with PAC)	Whang et al. (2004)

Table 2.2 summarizes several studies that utilized PAC in treating wastewater. The table reveals a higher percentage removal of COD was achieved when PAC was added to an anaerobic high salinity synthetic sewage in the study conducted by Vyrides et al. (2010). This was explained by the formation of biological powdered activated carbon (BPAC). BPAC can form when PAC is added intermittently to an MBR (Li et al., 2005, Vyrides et al., 2010). A biofilm forms on the PAC and that transforms it into BPAC-sludge, which enhances the pollutant removal as the microorganisms on the biofilm, can biodegrade the pollutants that had previously been adsorbed by PAC. There are three major advantages to BPAC. Firstly, it increases the efficiency of substrate removal. It also improves the activated sludge filterability, and reduces the adverse effects of toxic chemical species on biomass through adsorption. (Ng et al., 2006). In order for the biofilm to be stable fresh PAC must be added to the reactor based on the wasting rate of the system (Li et al., 2005). This, constant addition of new PAC was incorporated into the current study to simulate a stable BPAC to achieve higher removal rates.

The solids residence time (SRT) is an important parameter that significantly influences the performance of PAC-assisted MBRs. From Table 2.2 it can be observed that SRTs ranging from 20 days to 150 days have been tested (Tian et al., 2008; Lesage et al., 2008; Aquino et al., 2006). More membrane fouling was evident at lower SRTs of 10 days (Ng et al., 2013) when compared to 30 days (Ng et al., 2006). However, when PAC was added, fouling control appeared to be more efficient in cases of the shorter SRT (10 d), possibly due to the fact that at shorter SRTs required fresh PAC to be added more frequently, so active PAC was always present in the mixed liquor in adequate amounts. Similarly, Ma et al. (2014) operated 2 reactors, one at an SRT of 30 d and another at a longer SRT of 180 d. They reported that at the shorter SRT, the frequent replacement of PAC led to higher removal of low molecule weight (<5 kDa) effluent dissolved organic matter (DOM), which also exhibited higher hydrophobicity. In conclusion, both these studies indicated that a smaller SRT is ideal for PAC addition as fresh PAC will be added based on the wasting rate.

Based on Table 2.2, it can be observed that most of the studies (except study 3) were completed in an aerobic environment. Only one anaerobic study (Aquino et al., 2006) treated WW and this was conducted with synthetic WW as opposed to raw WW. The other two anaerobic reactors treated high salinity synthetic sewage (Vyrides and Stuckey, 2010),

municipal solid waste leachate (Trzcinski and Stuckey, 2010) and swine wastewater. There is a clear need to investigate the effect of PAC when treating raw DWW and based on the current studies PAC could assist in generating higher permeate quality.

In summary, it is evident that PAC addition has been found to improve COD removal in several studies, although in some cases the improvement was minor, especially for WWs with low concentrations of biodegradable compounds (Ying and Ping, 2006, Munz et al., 2007, Liu et al., 2007, Lesage et al., 2008, Remy et al., 2010). In addition, it was observed that PAC addition to MBRs yielded a better permeate quality when the influent had high COD levels and salinity, even for leachate streams (Vyrides and Stuckey, 2009, Trzcinski and Stuckey, 2010). Overall, it was concluded that a majority of the studies support the addition of PAC to enhance membrane performance.

Chapter 3 – Methodology and Materials

3.1 Test Plan

The purpose of this research project was to explore the effect that powdered activated carbon (PAC) had on the performance of AnMBRs treating WW in psychrophilic conditions. The objective was to determine if PAC addition affects anaerobic digestion and membrane performance under these conditions, and to identify possible relationships between these factors. In order to address these objectives, three lab-scale reactors were set up to treat raw wastewater.

Techniques and gaps identified in the literature review informed the design of the experiments, specifically in the areas of the influent used, the operating temperature, and the fouling control method employed. It was found that a majority of the reviewed studies used synthetic WW but raw WW was deemed to more accurately represent field conditions including seasonal variations. Thus, raw WW was used as the influent in the present study. In addition, a majority of the studies investigated AnMBRs and MBRs at mesophilic temperatures and measured %COD removal. As a control, one of the reactors in the present study was kept at 24°C and %COD removal was established as one of the performance indicators to be measured, allowing a comparison with prior research. Further, a practical and effective fouling control method was selected based on prior studies, and the method is described in section 3.2.3 of this chapter.

3.1.1 Outline

Three bench-scale reactors with an SRT of 30 days and an HRT of 0.4 days were set up in parallel and a comparative study was conducted in two phases. The aim of the first phase was to establish a baseline and to evaluate the effect of temperature on the system. The aim of phase 2 was to determine the effect that PAC has on the bioreactors and membranes. In phase 1, two of the reactors were set at a low temperature (10°C) and the third reactor was set at room temperature of 24°C and was treated as the aforementioned control. On day 214, the temperature of reactor C was lowered to 10°C and on day 241, PAC was added to all three reactors. Three complete SRT cycles (90 days total) were required to

reach the new quasi-steady state and phase 2 began on day 331. Figure 3.1 summarizes the two phases (with a transition between them) and the days that changes were incorporated.

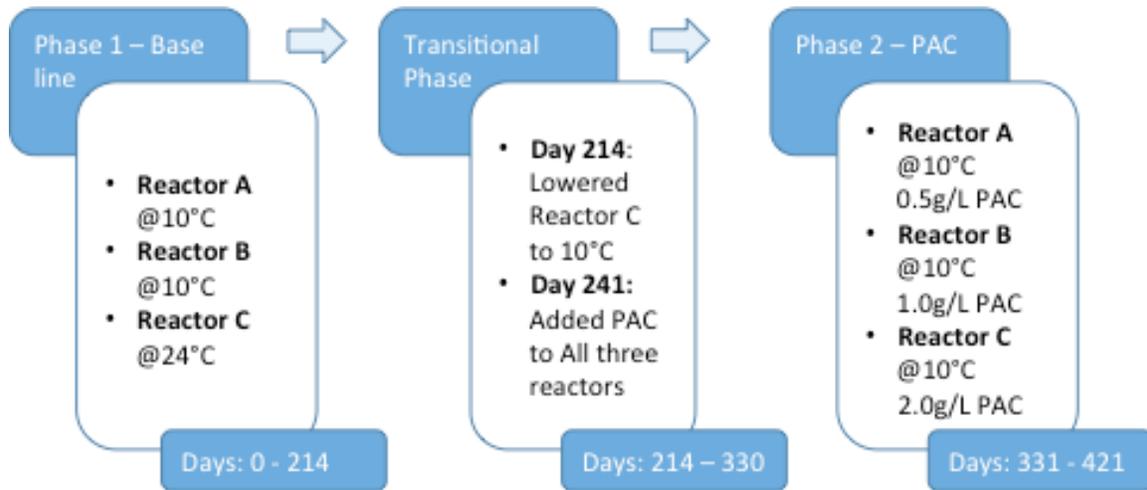


Figure 3. 1: Test plan

During phase 1, the three reactors were transferred from a previous study where they were operated with an SRT of 30 days and an HRT of 0.4 days. This SRT was maintained by wasting 133 ml WAS per day. The established SRT and HRT resulted in consistent, reliable and high permeate quality and were within the range of HRT's demonstrated in the literature review to be sufficient at psychrophilic temperatures. These parameters were thus continued from the prior operation of the reactors and maintained throughout all phases.

The reactors had been idle for some time before the present study began, so the initial task of phase 1 was to restart the reactors and reestablish a steady state. One reactor was set at 24°C and another at 10°C, both without PAC addition. The reactors at 10°C were insulated in a chilled water jacket with constant circulation of cold water. The reactor at 24°C was set up on a lab bench. In order to ensure that the temperatures were constant all reactors were monitored with a thermometer probe inside the reactor though out the day. Any fluctuations in the temperature were addressed by adjusting the temperature in the room or the water jacket. In order to start all the reactors at the same time, the 3rd reactor was also started and operated at 10°C. This was done so the transition into second phase would be smoother and all membranes would be the same age. Steady state was established after 90 days of operation.

Once sufficient data were collected for the baseline analysis, reactor C was converted to the lower temperature of 10°C. The temperature was allowed to stabilize for 26 days. Then, on day 241, PAC was introduced to each reactor in differing concentrations (0.5 g/L to reactor A, 1.0 g/L to reactor B, and 2.0 g/L to reactor C). The type of PAC used has been detailed in Appendix W. The PAC lost in the WAS was replaced every three days to maintain these PAC concentrations throughout the experiment. Phase 2 began on day 331, after having allowed three 30-day SRT cycles since PAC introduction in order to reach steady state.

3.1.2 Experimental Configuration

Figure 3.2 illustrates the configuration of a reactor. This was identical during all stages for all reactors. The only difference was that at 10°C there was an added water jacket around the reactors. The flow begins at the feed (orange line) and ends at the permeate (blue line). Fifteen liters of feed were collected from a main line and stored in two refrigerators at a temperature of 10°C. One fridge supplied Reactor A and B and the second supplied Reactor C, each had individual pumps collecting feed from the respective fridges. Both fridges were refilled daily. Each day, 10 L of sewage from the Regional Municipality of Waterloo was fed through a pump into the reactor. The reactors consisted of five-liter PVC cylinders containing the submerged hollow-fiber membranes. The feed was treated anaerobically in the reactor, and permeate was pulled through the membrane at a rate of 10 L/d by the pump to maintain an SRT of 30 days, 133 mL of WAS was separately wasted daily.

To maintain a consistent SRT and WAS level in the closed system, reactor pressure control was necessary. This was achieved by controlling influent into the reactor and regulating the membrane scouring system. A P-trap was attached to the membrane tank to relieve excess pressure and prevent overflow in the event of a permeate pump malfunction. The P-trap redirects any mixed liquor in excess of 4.5 L to the 10 L overflow tank, which excess could then be manually returned to the bioreactor to maintain the WAS level. The membrane sparging system involved a gas circulation pump, whereby nitrogen gas was flushed through the system to purge oxygen (initially and after any opening of the reactor), and nitrogen with any built-up methane was subsequently circulated through the reactor.

This added pressure was controlled by means of a Tedlar bag. The regulation of influence and of gas circulation maintained the consistency of the SRT and WAS level.

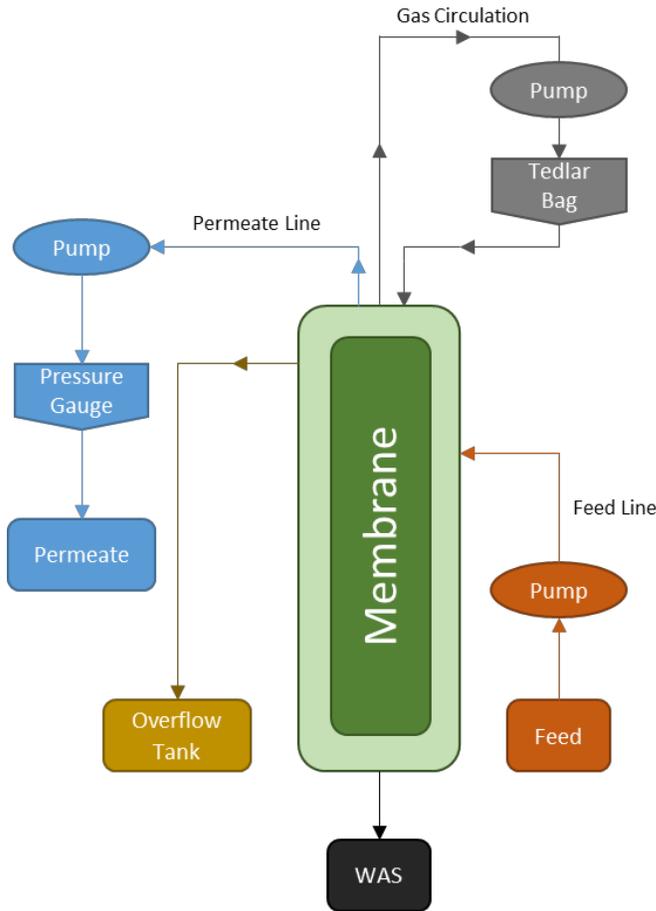


Figure 3. 2: Experimental Configuration

3.1.2.1 Apparatus

As shown in Figure 3.2, there were four main pieces of apparatus used: the membrane, the liquid pumps, the gas pumps and the pressure gauge. There were three reactors (A, B, C) with the same configurations as shown in the diagram. The membranes used were ZeeWeed® hollow fiber polyvinylidene difluoride (PVDF) membranes with a pore size of $0.04 \mu\text{m}$ and a surface area of 0.047 m^2 (GE, Guelph) The liquid pumps used were Masterflex peristaltic pumps (HV-77921-65) The same pump models were used for both adding feed and extracting permeate at a rate of 10 L/day. The air pumps used were KNF Laboport® series laboratory vacuum pumps (N 810 FTP). They were operated at a rate of 7.5 L/min to create a sparging effect for scouring the membrane. The pressure gauge

used was an Omega transducer (PX602-015GV). This was used with a timer to measure the TMP once per minute in order to capture and monitor it throughout the operation/rest cycle. The specifications can be found in Appendix W.

3.2 Operation of AnMBRs

The operation of the AnMBR began on 20 February 2017 and ended on 17 April 2018. Figure 3.3 charts the timespan of the major activities in the study relative to the starting date. In Phase 1, COD and SS concentrations in the reactor were deemed constant within 90-days. A similar approach was followed in Phase 2. After a steady state was reached, the reactor was operated for 90 days during which data collection was conducted according to the sampling plan listed in section 3.2.4.

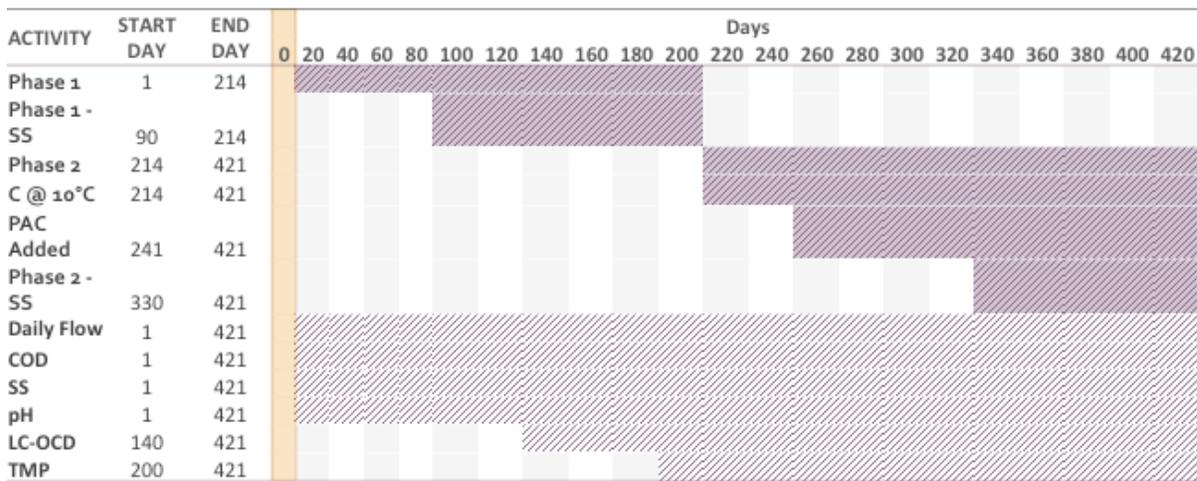


Figure 3. 3: AnMBR Operation Table

3.2.1 Influent for Anaerobic digestion

Raw domestic wastewater was used in this study. Ten liters of sewage were collected daily from the Regional Municipality of Waterloo sewage line that flows through the University of Waterloo. The WW was redirected from the sewage line to a holding tank installed for the use of the University's WW labs. The holding tank empties back into the sewage line via a purging pump controlled from the WW lab. The pump is normally engaged, but is disengaged in order to allow the holding tank to fill for lab use. A second outlet is used for sampling. Samples were collected and stored in a main fridge ready to be used in the study as needed.

3.2.2 Maintenance procedures

It was deemed critical to maintain the SRT, HRT, flux, and temperature constant throughout all phases consistent to get reliable samples. The daily 133 mL waste of WAS extracted from the reactor at 12:00 p.m. consistently to evenly space out the wasting. Daily permeate volume was monitored for consistency at 10 L/day. A decrease below 8.5 L was addressed within 12 hours, often by means of a cleaning procedure (see Section 3.2.3 below). Temperature fluctuations greater than 3°C were also investigated within 12 h. Such sudden changes in temperature were only expected in case of power failure.

3.2.3 Membrane Cleaning Procedure

There were two types of membrane cleaning procedures that were implemented throughout the experiment. These were maintenance cleaning (MC) and recovery cleaning (RC). Maintenance cleaning was conducted regularly to prevent downtime. Recovery cleaning was employed when maintenance cleaning was insufficient to bring the TMP back up or if the permeate generation was less than 5 L/day.

Maintenance cleaning:

There were three types of maintenance cleaning routines:

1. Backwashing with 250 mL of permeate at a rate of 16 mL/min. This was done every day to ensure that the membrane did not foul rapidly.
2. Backwashing with a cleaning solution (2 g/L of citric acid) at a rate of 16 mL/min. This was conducted once a week.
3. Rinsing the membrane with tap water to remove biofilm. This was done as needed, often twice a month. Over the duration of treatment, biofilm would accumulate on the surface of the reactor. This would cause the TMP to spike and the permeate volume to drop below 5 L/d. When this occurred, the reactor was turned off and opened up and the layer of biofilm was cleaned manually with running tap water.

Recovery Cleaning:

Internal fouling, rather than biofilm buildup, was deemed to be the cause of circumstances requiring recovery cleaning. This cleaning consisted of soaking the membrane in citric acid

at a concentration of 2 g/L for approximately 16 hours, rinsing it with tap water, then soaking it in sodium hypochlorite at a concentration of in 2 g/L for another 16 h. Finally, the membrane was washed again using tap water and placed back into the reactor.

3.2.4 Sampling Procedure

The raw WW influent, the WAS, and the treated permeate effluent samples were analyzed according to the sampling schedule detailed in Table 3.1. All samples were analyzed immediately according to the methods described in section 3.2.5.

Table 3. 1: Sample Schedule

Parameter	Type of Sample	Frequency	Starting
Flow	System	1/Day	Day 1
pH	Feed, WAS, Permeate	2/Week	Day 1
Temperature	Reactor	1/Day	Day 1
TMP	Reactor	1/minute	Day 200
COD	Feed, Permeate, WAS	2/Week	Day 1
SCOD	WAS	2/Week	Day 1
TSS	Feed, WAS	2/Week	Day 1
VSS	Feed, WAS	2/Week	Day 1
LC-OCD	WAS	1/Week	Day 140

TMP and LC - OCD was started later due to availability

3.2.4.1 Feed Collection

A volume of 200 mL of feed was sampled from the feed leaving the refrigerators for all reactors. Since reactor A and B shared a feed line, one feed sample was taken for both of them and a second sample was taken from the reactor C feed line. TCOD tests were conducted immediately. Simultaneously, 100 mL of the sample was kept in the homogenizer as preparation for the SCOD test.

3.2.4.2 WAS Collection

As explained in section 3.3.2, 133 ml of WAS was wasted from the reactor daily at noon. On test days, the sample was preserved in the refrigerator at 10°C instead of being wasted. All the tests outlined in Figure 3.3, except LC-OCD were conducted within 4 h of sampling. The LC-OCD samples were prepared and stored and were tested with 24 h of sampling.

3.2.4.3 Permeate Collection

The permeate accumulated in a PVC container over the course of operation. At the time of sampling the container was vigorously shaken, then 2 L were poured out and a sample of 200 mL was taken from the permeate container. The tests specified in Table 3.1 was done within 4 h of sampling.

3.2.5 Sample Analysis

Physical and biochemical properties we analyzed using conventional methods and according to the appropriate section of the Standard Methods for the Examination of Water and Wastewater (Eaton et al., 2005). Each sample was measured in duplicate and standard samples were prepared for all analyses.

3.2.5.1 TCOD

COD analysis was conducted according to Standard Method 5220 D (APHA, AWWA, WEF, 2012). To determine the TCOD of a sample, 50 mL of the sample was homogenized for 30 seconds. 2.5ml of the mixed sample was appropriated diluted and added to a vile containing 1.5 mL of COD digestion solution and 3.5 mL of sulfuric acid reagent. The vile was inverted several times and then placed in a 150°C preheated HACH COD reactor for 3 h. The samples were allowed to cool to room temperature, then they were measured at 600 nm using a HACH DR/2000 Spectrophotometer. Every day of testing, new blanks and calibration samples were also prepared and subjected to experimental procedures to produce a calibration curve.

3.2.5.2 sCOD

To determine the sCOD of a sample, 50 mL of the sample was centrifuged for 30 min and the supernatant was filtered through a Whatman Glass Microfibre filter (934-AH) with a pore size of 1.5 µm. Once 3.0 mL of sample was obtained, it was subjected to the same testing process outlined in 3.2.5.1 for TCOD.

3.2.5.3 SS

Total suspended solids and volatile suspended solids were measured according to the standard methods 2540D and E (APHA, AWWA, WEF, 2012). TSS was measured by filtering a set volume of sample though a Whatman Glass Microfibre filter (934 – AH) with a

pore size of 1.5 μ m that was previously weighed and dried at 450°C for at least one hour. The filtered samples were placed in aluminum dishes and, using tongs, were carefully placed in the oven at 105°C for 24 hours to dry. The increase in mass was the TSS mass. The sample was then placed in the oven at 550°C for an hour. The reduction in mass represented the VSS. All the transportation of the filtered samples were inside aluminum dishes carried out using a covered container to avoid an loss of sample during transportation though the lab.

3.2.5.4 pH

The pH of the samples was measure daily as outlined in Table 3.1 using as Omega PHB-600R pH Benchtop Meter.

3.2.5.5 LC-OCD

Initially, 50 mL of the sample was centrifuged for 30 minutes and the supernatant was filtered through a Whatman Glass Microfibre filter (934-AH) with a pore size of 0.45 μ m. These samples were then transported to the drinking water lab to perform the LC-COD analysis as per (Huber et al., 2011). LC-OCD separates molecules based on their molecular weight, size, shape and interactions with other molecules. 1 mL of sample was injected to into a phosphate buffer solution at a pH of 6.58, which formed the mobile phase which passed through the stationary column for molecule separation. The size exclusion chromatography column was connected to the UV detectors and organic carbon detectors. Based on the shape of the curves from the detectors the NOM was separated into biopolymers (BP), humic substances, building blocks, low molecular weight (LMW) acids and neutral fractions. The analyst provided the values.

3.2.5.6 TMP

The transmembrane pressure was measured using an Omega transducer (model, supplier, etc.). This was used with a timer to measure the TMP once per minute in order to capture and monitor it throughout the operation/rest cycles. The data were stored on...?

3.2.5.6 Cleaning Frequency

The duration each reactor ran before a recovery cleaning was required was measured. This was to substitute for the lack of TMP data during Phase 1. It was continued through to Phase 2 to maintain consistency.

Chapter 4 – Results and Discussion

The following chapter presents the results of the experimental testing described in Chapter 3. Section 4.1 explores the feed data, beginning with the raw data. This is then followed by Phase 1 and 2 results in sections 4.2 and 4.3, respectively, commencing with bioreactor properties followed by membrane responses.

4.1: Influent Characteristics

Water quality parameters such as TCOD, SCOD, TSS and VSS of the influent are discussed in this section. These data provide a platform to compare the effluent and WAS values that are described in the later sections of this chapter. As detailed in Chapter 3, there were two influent lines; line AB and line C, each feeding the respective reactors. Figures 4.1 and 4.2 display the COD and SS values, respectively, over the entire experimental phase for both lines. Analyzing them separately provided an understanding of factors that might influence reactor conditions and performance.

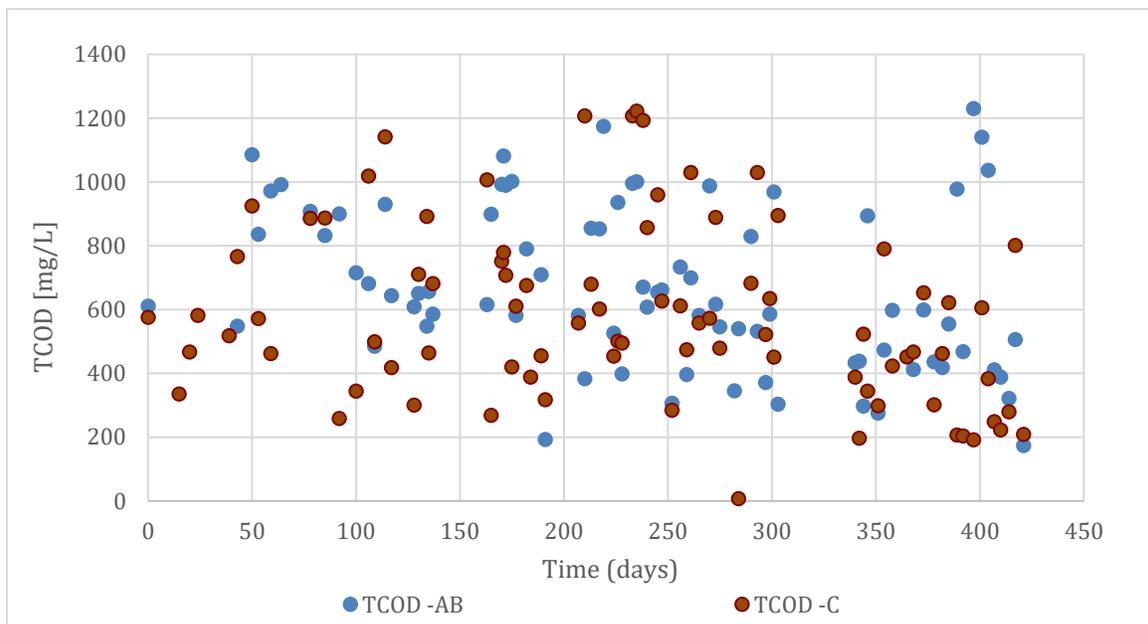


Figure 4. 1: TCOD Concentrations in the influent during the research period

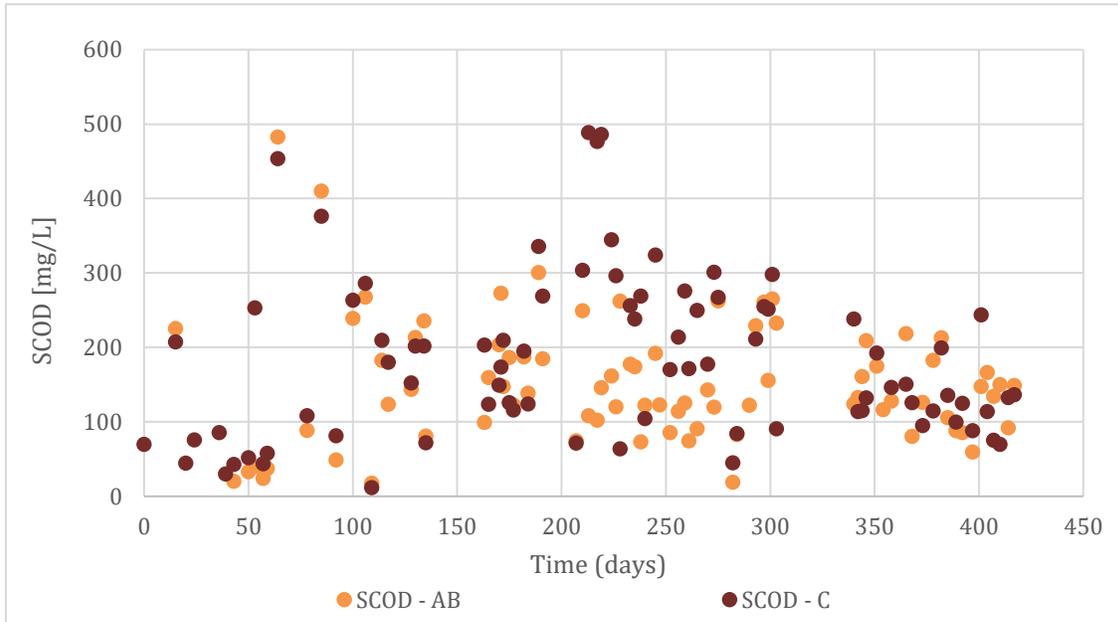


Figure 4. 2: SCOD Concentrations in the influent during the research period

Observing the TCOD in the feed provided a baseline for COD balancing, as this amounted to the total COD entering the reactors. In addition, this indicated how the concentrations inside the reactors could be affected. Figure 4.1 illustrates the feed concentration of TCOD that was delivered through each line over the course of testing. The sampled influent values were within the range of 200 mg/L to 1200 mg/L, with mean values of 755 and 653 mg/L for reactors AB and C, respectively. Typical WW TCOD values range from 350 mg/L to 1200 mg/L depending on the type of WW (Metcalf & Eddy, 1991). The influent had a high range of data values despite the constant feed source. Hence, it was concluded that a high level of fluctuation in TCOD levels could be expected in the reactors.

The SCOD entering the reactor is indicative of the COD that might persist to the permeate and of the biodegradability of the wastewater, as SCOD tends to consist of readily biodegradable COD. Figure 4.2 illustrates the SCOD levels entering each reactor throughout the entire duration of sampling. The SCOD concentration in the feed streams ranged from 200 mg/L to 480 mg/L with the majority (>80%) of the SCOD values reading below 350 mg/L. The average values were 144 and 160 mg/L in the AB and C lines, respectively. A portion of the SCOD was expected to be non-biodegradable (nbSCOD), and hence, persist to the permeate stream. The biodegradable SCOD would be expected to degrade to methane in the bioreactor. Thus, the concentration of SCOD was expected to have a major influence on permeate quality and biogas generation.

The TSS entering the reactors were expected to impact the concentrations inside the reactors. The TSS is comprised of VSS and FSS, the latter which consists of non-biodegradable minerals that will accumulate in the reactor and be removed only in the process of sludge wasting. The former VSS is comprised of biodegradable (bVSS) and non-biodegradable (nbVSS) components. bVSS will be removed through hydrolysis to produce methane, while nbVSS will accumulate in the mixed liquor and might contribute to membrane fouling through cake formation on the membranes. Figure 4.3 displays the SS for both streams of influent with TSS shown on the primary axis, and VSS plotted on the secondary axis. A majority (>80%) of the TSS values were below 800 mg/L which is slightly greater than that reported for conventional WW (600 mg/L) (Metcalf & Eddy, 1991). It was anticipated that the elevated suspended solids concentrations in the feed could lead to elevated TSS levels in the AnMBRs if they were not hydrolyzed in the reactor.

The entire experimental duration was 435 days and it consisted of two phases where the three reactors all received the same feed. However, it was suspected that there could be variation in the feed composition due to factors such as accumulation of solids in containers or change in composition between phases. To check for this t-tests were completed to compare properties between the two feed lines and between the two phases as subsequently described.

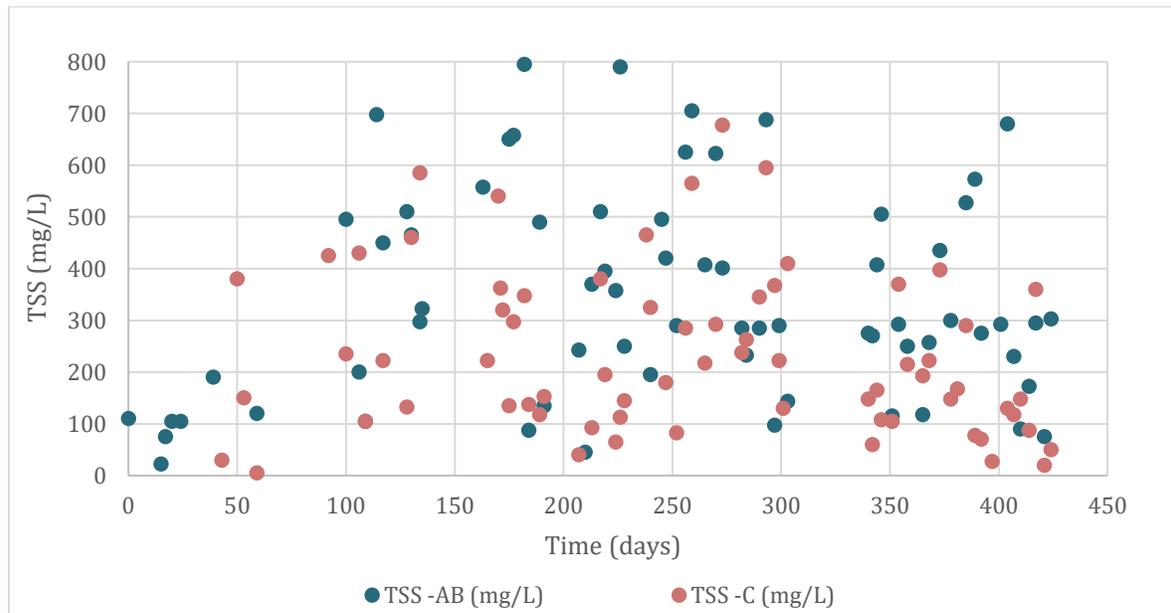


Figure 4. 3: Concentration of TSS in the influent during the research period

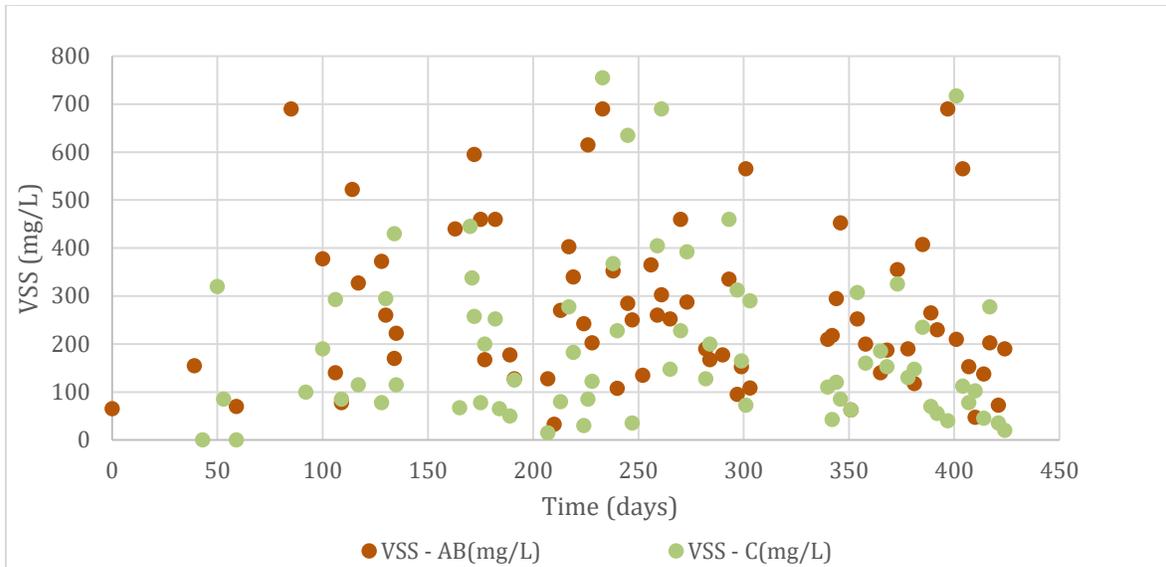


Figure 4. 4: Concentration of VSS in the influent during the research period

4.1.1 Comparison of feed streams

It was deemed to be important to determine if there was a significant difference between the influent characteristics of the two feed lines over both phases, as differences in feed composition would be reflected in the AnMBR performance. The influent concentrations of TCOD of the lines to reactors AB and C were compared using a t-test at a 95% confidence interval (Appendix A1) and the concentrations were found to be indistinguishable from each other. This was expected as both feeds were taken from the same source and fed into each reactor within 12 hours of collection. This permitted direct comparisons to be made between reactors in subsequent chapters.

Similarly, the TCOD concentrations observed in phase 1 (Day 90 – 214) and phase 2 (Day 331 – 421) was compared with t-tests once steady state was reached. The steady state was determined as the day when all three reactors in all measured parameters were not demonstrating significant fluctuations. This was determined as Day 90 for phase 1 and Day 331 for phase 2. The determination of steady state is explained later on in detailed. It was found that the two data sets were statistically different (Appendix A2). The difference in feed quality between phase 1 and phase 2 was attributed to seasonal variation in wastewater properties. Phase one was conducted from 23rd May – 22nd Sept with an average TCOD of 738 mg/L (SD = 228) which was significantly greater than phase two values which had an average TCOD of 511 mg/L (SD = 273) over the period 20th Jan – 17th April.

Snowmelts that occur during the winter months could add water to the sewer lines hence diluting the feed. This could be a possible explanation of the decrease in TCOD levels in phase 2, as it was conducted during winter. This means that the reactor concentrations could not be directly compared between phases. It also implied that higher level analyses such as COD balances need to be conducted to allow for inter-phase comparisons.

4.2 Phase 1 Results

In this phase, AnMBRs A and B were operated at 10°C and C was operated at 24°C. The aim of the first phase was to establish baseline performance without PAC addition and to evaluate the effect of temperature on the systems. The parameters that were evaluated in this phase addressed bioreactor performance. They included TCOD, SCOD, TSS and VSS in the WAS of each reactor, and the TCOD of the permeate. A COD mass balance was conducted and methane generation was estimated from this analysis. The membrane performance was evaluated based on the time between cleanings of the membranes and LC-OCD data that were available for a portion of phase 1. A sample of the raw data are presented in Appendix B.

4.2.1 Validation

It was deemed important to validate the lab data to identify any systematic errors, random errors or blunders. The data were subjected to two main checks. It was expected that the concentration of SCOD in the WAS should be greater than the concentration of the TCOD in the permeate, as the pore size of the membrane (0.04 μm) was smaller than the filter paper for the SCOD tests (1.5 μm). Furthermore, it was expected that the TCOD of the WAS would be proportional to the VSS measurement as this stream mostly consists of suspended solids. A typical ratio for bacterial biomass is 1.42 gCOD/g VSS (reference). Thus, theoretical TCOD concentrations were calculated for the WAS from the measured VSS values and compared to the measured TCOD in the WAS. These checks were applied to each reactor as presented in Appendix J; all anomalies were eliminated and the validated data were employed in subsequent analyses.

4.2.2 Analysis and Discussion

4.2.2.1 Bioreactor

The bioreactor data were initially assessed to determine when the reactors had reached a dynamic steady state. Figure 4.5 shows the TCOD levels inside all three reactors over the duration of phase 1. From the figure, it is evident that all the reactors had not reached steady state in the first 72 days as the concentration of COD was still increasing in all three reactors. Although it could be argued that Reactor B and C reached steady state earlier as the concentration is around 15,000 mg, however reactor A has varying concentrations. Thus, in order to maintain consistency, steady state was determined to be post day 78 as reactor A also steadied out by then. The increase in concentration was attributed to the growth in biomass as all three reactors were switched off for two weeks prior to the research. After day 78, all three reactors continued to fluctuate, but less drastically and with no discernible trend. Typically reactors reach steady state within three SRT cycles (Metcalf & Eddy, 1991) so for analytical purpose it was deemed that steady state was achieved from day 90 onwards on the basis of relatively constant concentrations and the fact that the reactors had been operated for three SRTs.

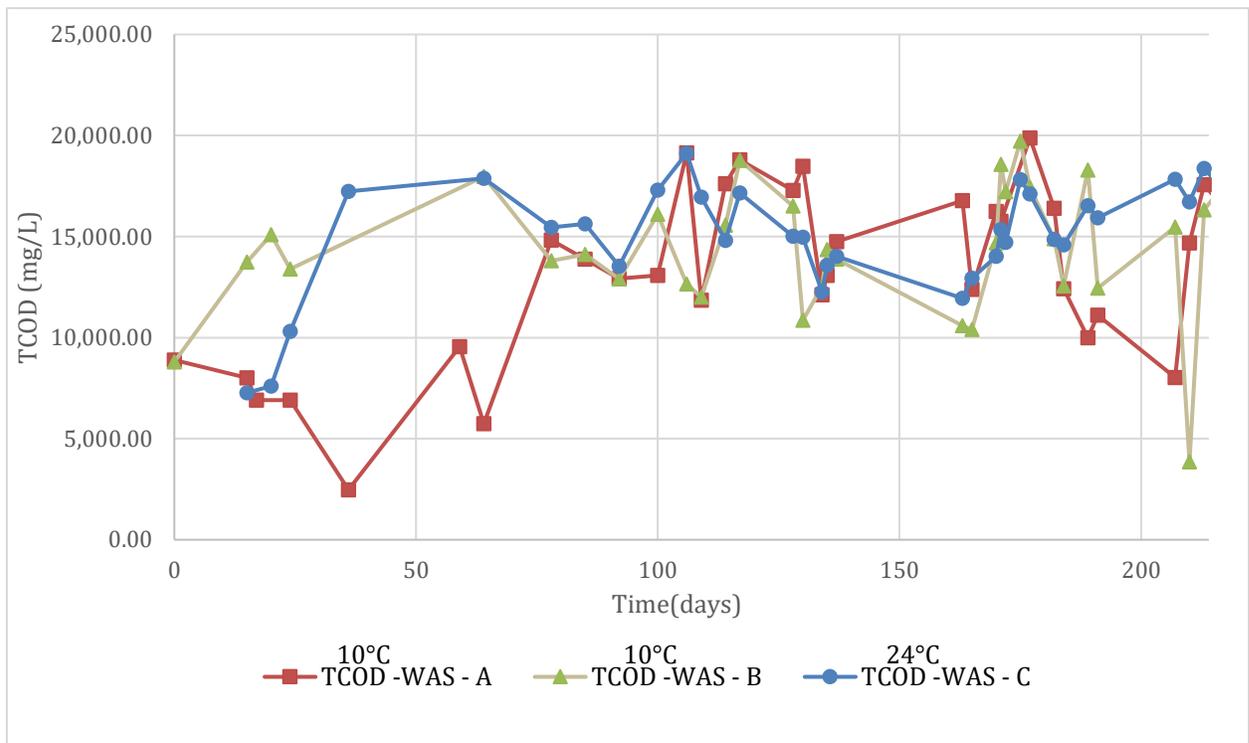


Figure 4. 5: TCOD concentration WAS for all three reactors

TCOD consists of bCOD and nbCOD which impacts how COD will be distributed throughout the system. It can be expected that the rbCOD portion of the bCOD will be rapidly degraded, whereas the sbCOD will initially be degraded by hydrolysis and at a slower rate. In contrast, the nbsCOD of the portion of the nbCOD will not be treated and will end up in the effluent, while the nbpCOD will accumulate in the bioreactor and leave in the WAS stream. The TCOD concentrations in the WAS were expected to be mainly comprised of nbpCOD and sbCOD. A high level of the latter would indicate that hydrolysis was not happening as rapidly, resulting in an abundance of both colloidal and particulate sbCOD. These can increase fouling in reactors though both cake and pore fouling. The average TCOD values (Figure 4.5) for reactors A, B and C were determined to be 14800, 14300 and 15500 mg/L with standard deviations of 3040, 3730, 2810, respectively. Hence, the reactors operated at lower temperatures appeared to have an average lower level of TCOD present in the WAS. The data were subjected to an ANOVA test at a significance of 95% as shown in Appendix S, which showed a significant difference. This led to conducting a Tukey test which indicated that the difference was between reactor A and C, but not between A and B, and B and C. Thus, the analysis was not effective in differentiating the reactors and so another approach was considered. The data for reactor A and B were combined and a t-test was conducted to identify a significant difference in the two temperatures. At the warmer temperature (24°C), there was a higher concentration of TCOD in the WAS. This suggested that there was more sbCOD or nbpCOD in the WAS of the warmer reactor as the nbpCOD was expected to be similar in all three reactors due to identical feeds. This was unexpected as lower levels of sbCOD were expected at warmer temperatures.

To further evaluate this observation, the accumulation of TCOD in the WAS was assessed through cumulative mass plots (Figure 4.5) and linear regression models. The gradients of each line were estimated to quantify the mass of COD accumulated per day. Both reactors at 10°C had a gradient of approximately 1700s g/d whereas reactor C at 24°C had a gradient of approximately 2000s g/d, which was approximately 1.2 times greater, throughout phase 1. A hypothesis test was conducted on the coefficient of the regression model at a significance level of 0.05 as shown in Appendix C. The test revealed that the coefficients were significantly different. Assessing the mean, the cumulative data and statistical analysis, it was concluded that less TCOD was present in the WAS at the cooler temperatures. This suggested that there was more sbCOD in the WAS of the warmer reactor, as the nbsCOD and nbpCOD were expected to be similar in all three reactors as the feeds

were identical. This means that hydrolysis was likely the limiting factor at warmer temperatures; this was not intuitive or expected.

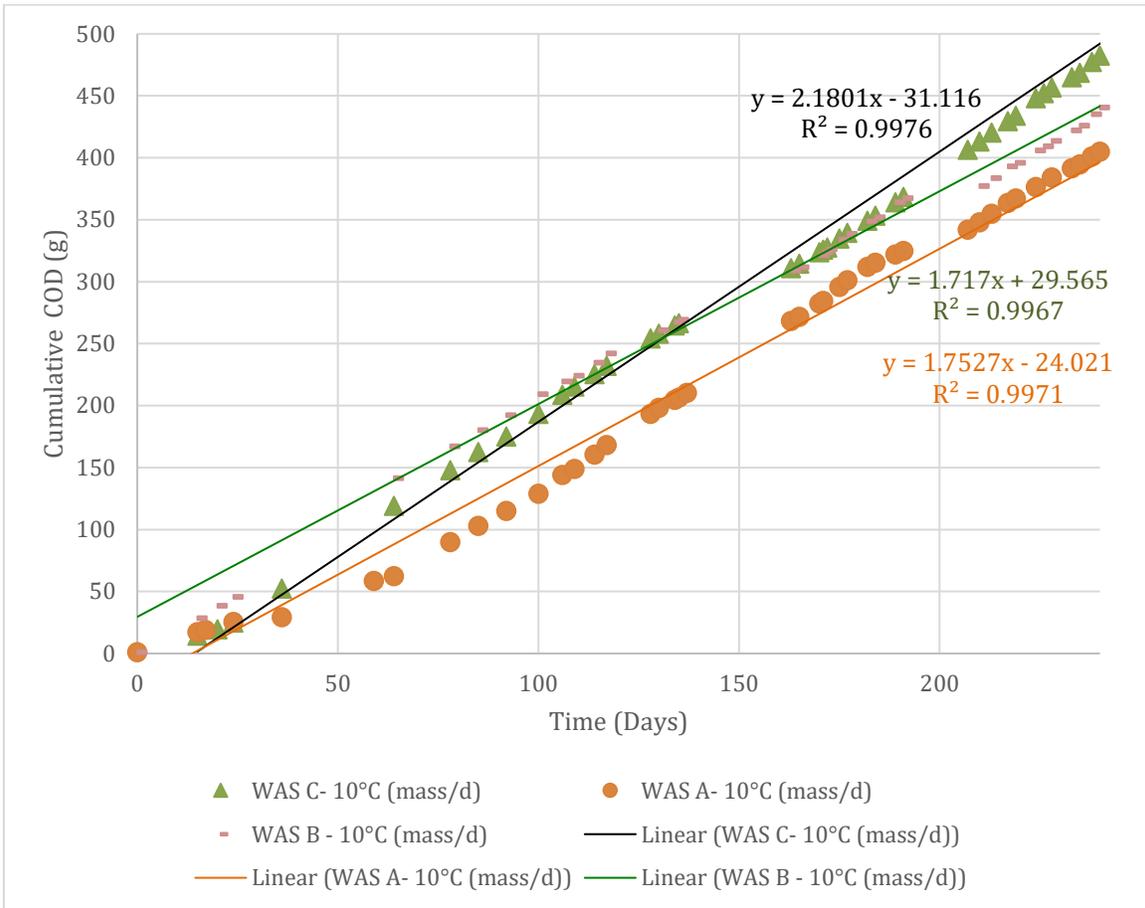


Figure 4. 6: Cumulative TCOD in WAS in phase 1 for all three reactors

SCOD consists of nbsCOD, colloidal COD and rbCOD. It was expected that the rbCOD would be biodegraded rapidly and was assumed to be negligible in the WAS. nbsCOD could end up in the permeate and can cause fouling of the membrane depending on its size. The colloidal COD could also be a foulant and deteriorate membrane performance, so for these reasons it was measured and investigated. Figure 4.7 illustrates the average SCOD concentration versus time in the reactor contents in phase 1. There was constant fluctuation throughout the whole phase, but generally all three reactors had concentrations that were in the range of 10-300 mg/L of SCOD. In order to keep the analysis consistent throughout the phase, steady state was set from day 90 onwards for all COD analysis and the averages were calculated. Reactors A, B and C had average concentrations of 271, 153, and 112 mg/L of SCOD present in the WAS. The data for all three reactors were compared with an ANOVA

test as shown in Appendix C2 at a significance level of 95%, which showed that there was a significant difference, and hence a Tukey test was conducted (Appendix C2). This revealed that there was a difference between reactor A (at X°C) and the other two and that the concentrations were higher in reactor A. It was assumed that the nbsCOD was not being affected by the reactor, so it would be consistent though all the reactors. The results suggest that the rbCOD was degraded differently in the reactors. This was unexpected as both A and B were under the same operating condition (including temperature-10°C) so the rates of biodegradation were expected to be similar. The different SCOD concentrations in these reactors and this may have been an experimental artifact.

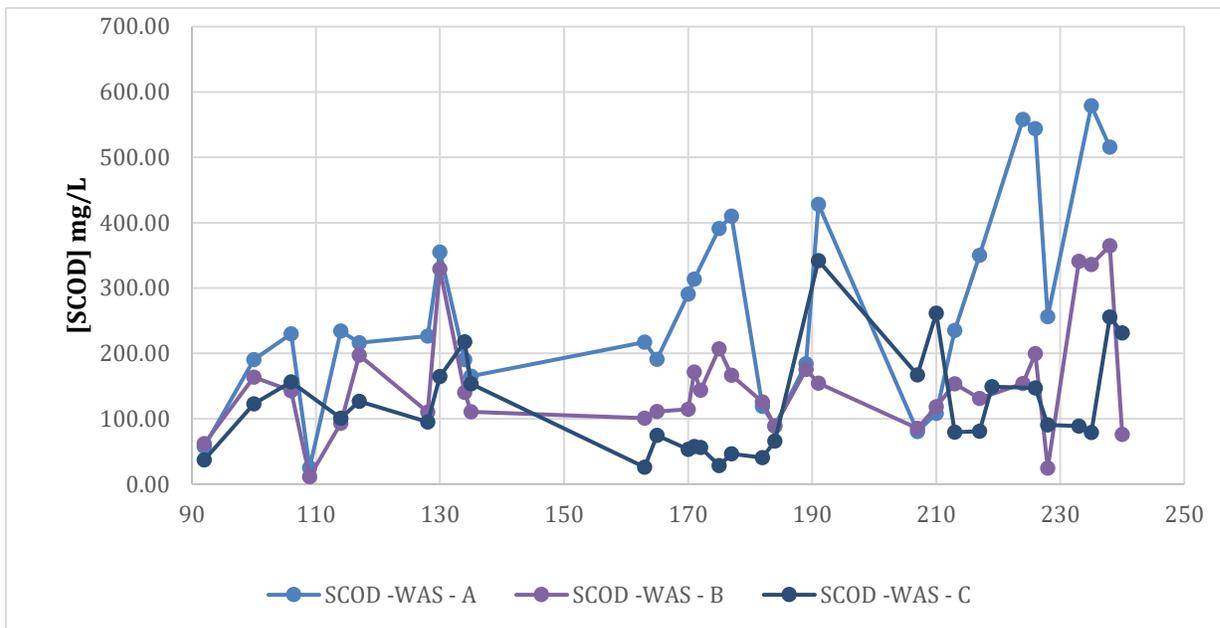


Figure 4. 7: The SCOD concentration of all three reactors in Phase 1

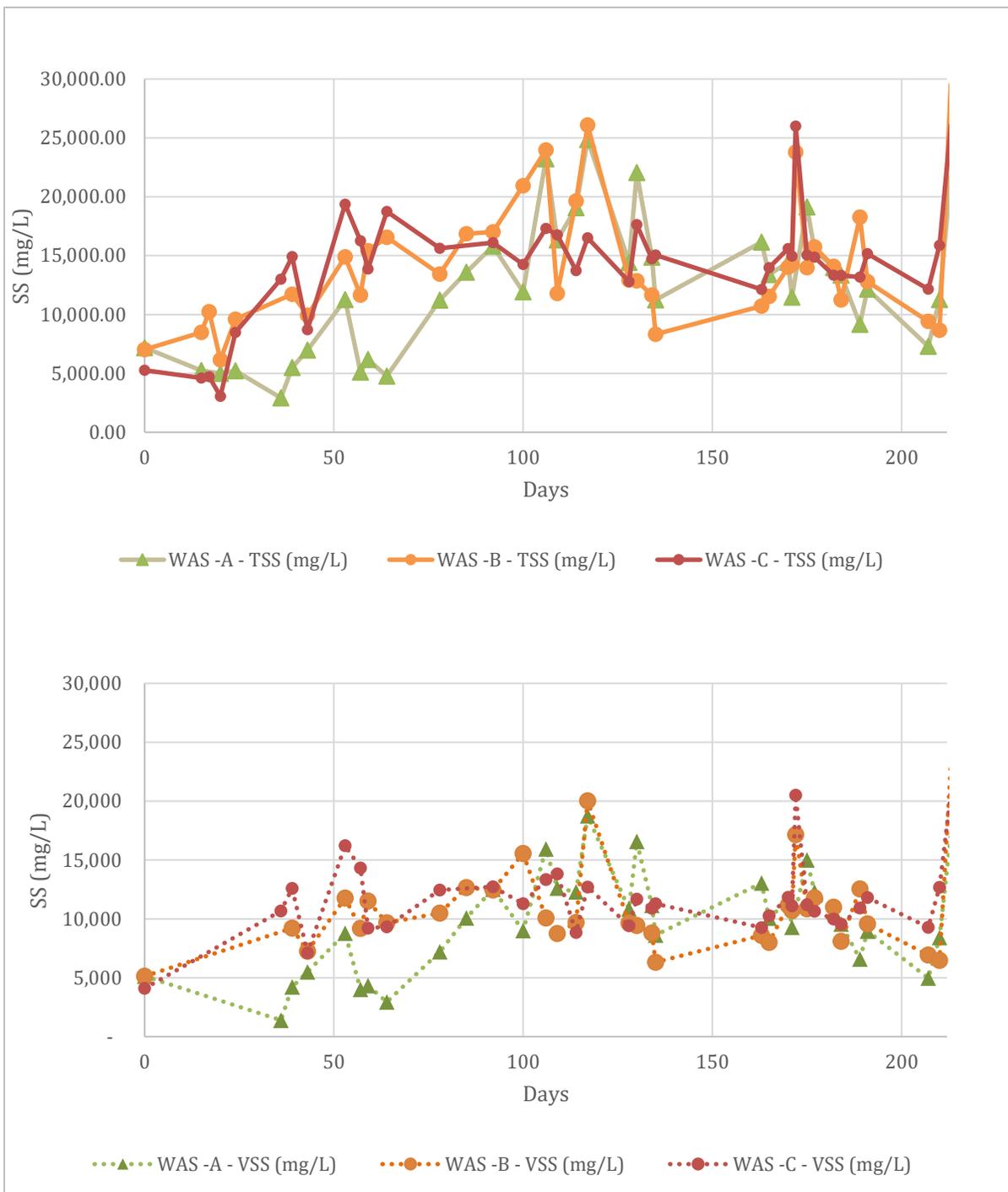


Figure 4. 8: Suspended Solids for All three reactors in phase 1

TSS and VSS in the bioreactors reflect the net impact of growth, endogenous decay and accumulation of feed particulates due to the difference between hydraulic and solid residence times. Figure 4.8 illustrates both parameters for all three reactors versus time. The TSS concentrations were low (approx. 5,000 mg/L) at the start of experimentation, climbing to approximately 24,000 mg/L on day 106 with several fluctuations over this period. In general, after day 78, the three reactors generally fluctuated in the range of

10,000 to 20,000 mg/L for TSS levels. The reactors were assumed to have reached steady state at this point, which corresponded to the TCOD data. As mentioned earlier the TCOD levels reached steady state at the around 70 days and steady state was taken from day 90 onwards to be safe. Since the SS data has reached steady state before 90 days it is still possible to use all the data. Hence, steady state was set from day 90 onwards for consistency in addition it also corresponded to 3 SRTs.

The TSS and VSS responses are indicative of particulate organic concentrations and includes the bacteria present in the WAS along with other particulates. The values presented in Figure 4.8 were employed to calculate average values after steady state was established, with reactor C having the highest average TSS of 17,918 mg/L followed by B at 17,060 mg/L and lastly reactor A with 16,497 mg/L. Application of statistical analysis indicated that the values were not significantly different as shown in Appendix D. However, A and B had lower average VSS concentrations of 12,100 and 12,243, respectively, whereas C had a higher VSS of 14,893mg/L. An ANOVA was completed to determine if there was a significant difference between the three reactors at a significance of 95%, and one was identified as shown in Appendix D. A Tukey test was used to identify that reactor C at a warmer temperature (24°C) had a significantly higher concentration of VSS. A possible explanation would be that the temperature enhanced the growth of the bacteria in the reactor resulting in higher VSS concentrations.

Permeate

The permeate samples were collected and analyzed similar to the WAS samples for COD concentration. The COD concentrations in the permeate provided a measure of the overall treatment efficiency of the system. The quality of the permeate provides information as to whether further treatment process would be required before discharge. Figure 4.9 shows the permeate COD concentrations and it is evident that substantial fluctuations were present for the first 85 days. After this period the results became steadier, suggesting that steady state was reached by day 92. The data were summarized in terms of mean, median and standard deviation values as shown in Figure 4.10, once steady state was reached. From this figure it can be seen that reactors A and B appeared to have higher COD values compared to C with very similar standard deviations.

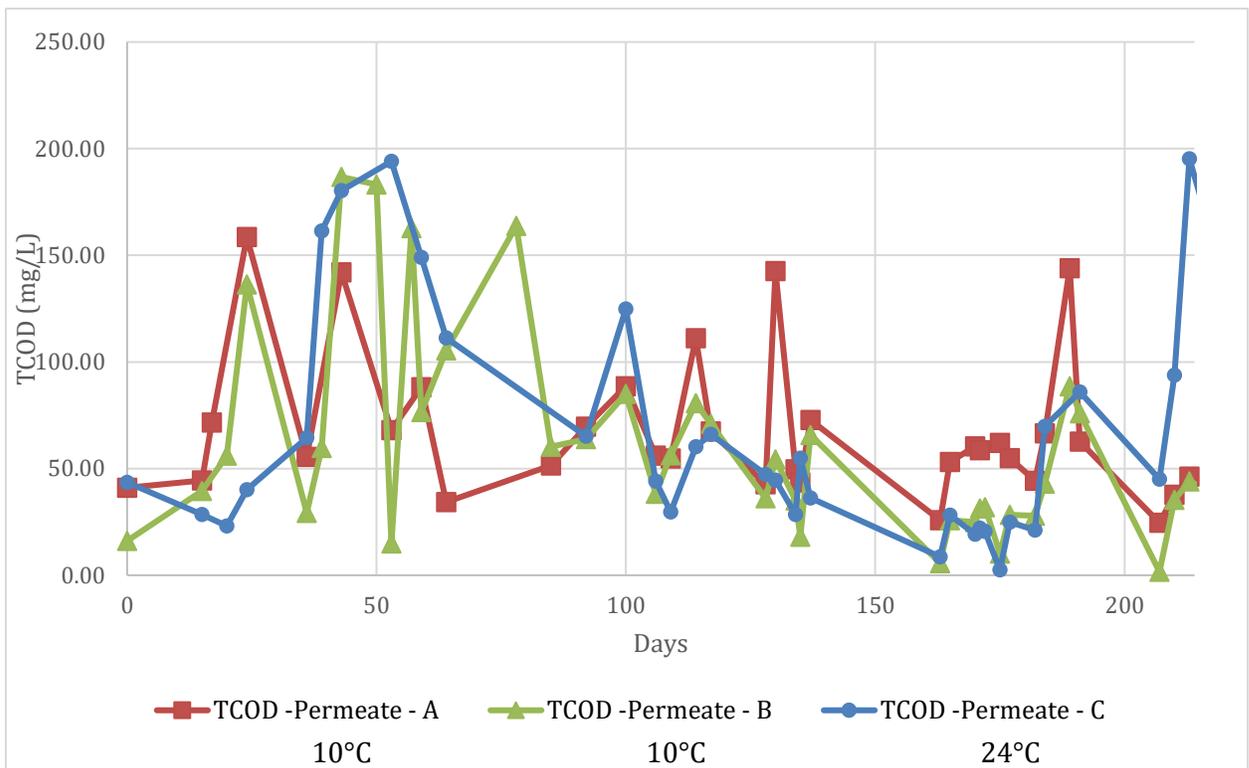


Figure 4. 9: COD concentrations of the three reactors in phase 1

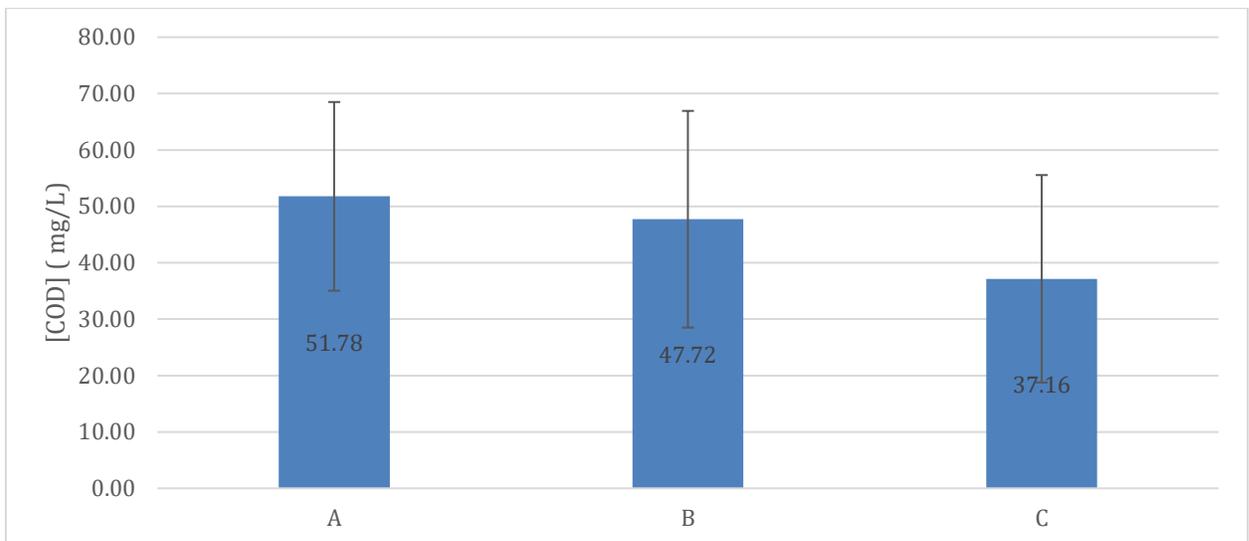


Figure 4. 10: The Average and standard deviation of permeate quality in all three reactors

An ANOVA was conducted at a significance level of 95% to determine whether the concentrations were different, as shown in Appendix E. This test indicated that there was a significant difference as the F - critical was less than the F-calculated. However, when a Tukey test was completed to identify where the difference was, it showed that there was a difference between A and C but not B and C. To address this, the data for A and B were combined as there was no significant difference between A and B (appendix E) and a T-test

was completed in comparison to C at a significance of 95%. This showed that reactor C had a significant lower concentration of COD, indicating that permeate quality decreased when the temperature decreased. This finding is consistent with the model that methanogens were responsible for attaining low permeate COD concentrations by removing VFAs. Methanogen growth could be expected to be reduced at low temperatures.

COD Fate

An analysis of the fate of COD in the reactors was conducted to assist in identifying the limiting step of anaerobic degradation in the reactors. This involved completing a COD balance to determine the fate of COD in each reactor. In this analysis the COD used for sulfate reduction was calculated for each reactor as shown in Appendix F. Once all the COD fates were calculated, the COD present in methane was estimated as the unaccounted COD as shown in Appendix G. Appendix G presents plots of the cumulative mass of COD in each reactor. These were summarized in Figure 4.11. In Figure 4.11 it can be seen that the fractions of the COD leaving the reactors as methane appeared to be high and unrealistic. The calculated methane fractions were converted to a volume of biogas that would be generated daily, and they were found to be greater than several liters, which was not observed in the lab. The elevated methane yields were calculated as the missing COD in the mass balance and hence any inaccuracies were incorporated into this portion. Thus, when analyzing the COD balance results it was concluded that the information on the non-methane fates was more accurate. Reactor C, which was operated at the warmer temperatures of 24°C had, on average, 5.6% higher portion of COD in the WAS, as compared to reactors A and B and a smaller portion of COD in the form of permeate and methane. This suggested that less of the organics were hydrolyzed and as the COD in the form of methane and permeate fractions were 4.3% and 1.6 % lower in reactor C. This is an indicative measure that hydrolysis could be the limiting process in the reactor, and as previously mentioned, this was not expected.

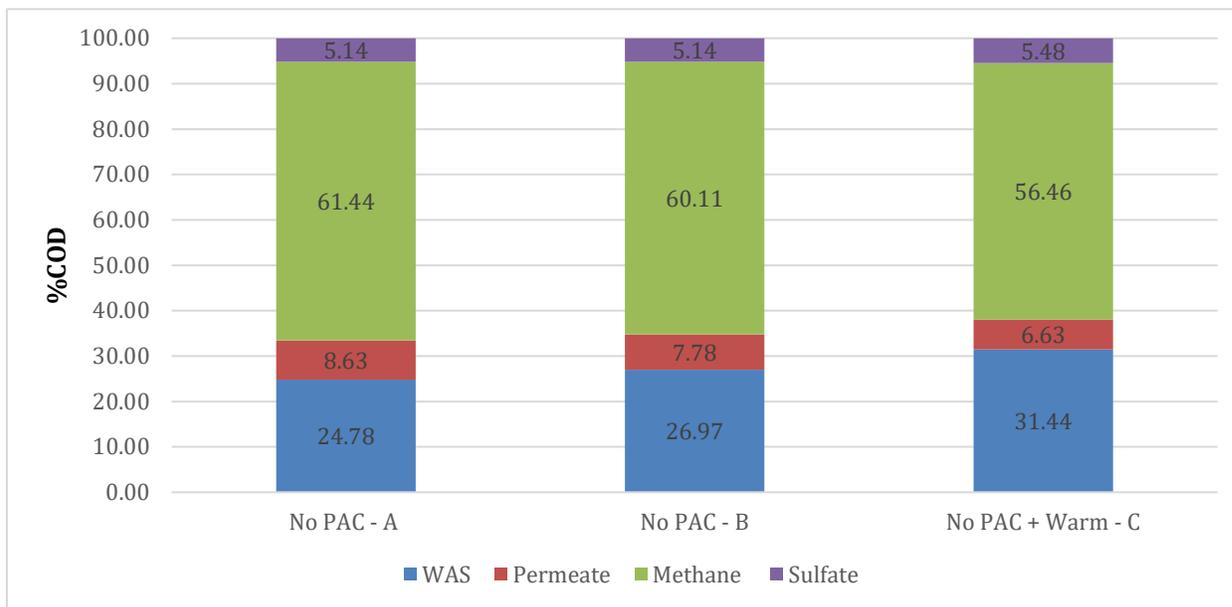


Figure 4. 11: COD distribution in the three reactors during phase 1

4.2.2.2 Membrane responses

LC-OCD

Liquid chromatography – organic carbon detection (LC-OCD) is an analytical technique for identifying and quantifying natural organic matter (NOM) constituents in water samples including wastewater. These can act as foulants in an AnMBR through various ways two main forms include partial entry into membrane or accumulation as part of the biofilm. Monitoring for LC-OCD fractions began on day 140 of testing. NOM constituents can be generated, biodegraded or remain unaffected in the AnMBR. If they were degraded or generated in the reactor it could be expected their removal would be affected by temperature as increases typically enhance microbial activity. Biopolymers, humics, and building blocks were investigated in detail as they are potential foulants (Zheng et al., 2017). The concentrations of the LMW substances had substantial fluctuations in the testing. For example, the LMW concentrations ranged from 1 µg/L - 94496 µg/L over the duration of the testing. The high variability suggested that these data were unreliable and thus has been presented in Appendix H but not explored further.

Humics have been previously reported as foulants responsible for bio-fouling and biofilm formation especially in drinking water. (Thekkedath, 2010) Thus similar results can be expected. However it is possible that since there are more foulants in WW the effect of humics might not be as significant. The concentration of humics fluctuated with time through phase 1 as can be observed in Figure 4.12. The humics concentration in reactor A increased in the initial four weeks and was notably higher than the other two reactors, thus resulting in a larger mean humic material concentration. From day 192, Reactor A concentrations followed the patterns in the other two reactors. The cause of the elevated levels of humics in reactor A in the early stages is unclear. It is unlikely that this was due to experimental issues as the rise was gradual, as was the decrease. Overall, there was no clear pattern or difference between the reactors in this phase. It can be seen that the responses for all three reactors were fluctuating suggesting that all three reactors increased and decreased at the same time but with varying magnitudes. Reactors A, B and C had average concentrations of 6760, 4600, and 4420 $\mu\text{g/L}$ respectively with corresponding standard deviations of 2400, 849, and 1419. An ANOVA with a significance level of 95% revealed that there was not a significant difference in humics concentrations between the three reactors as shown in Appendix I. Hence, it was concluded that the mechanisms contributing to the humics concentrations were not affected by temperature.

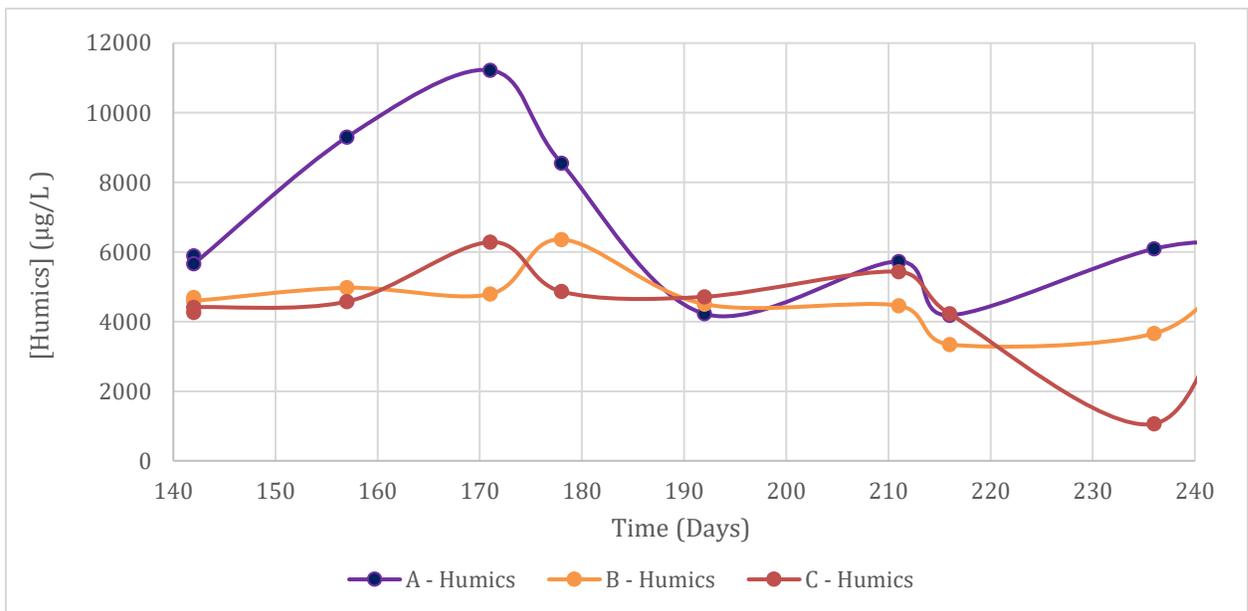


Figure 4. 12: Humics concentration in phase 1

The concentrations of biopolymers that were observed in Phase 1 are displayed in Figure 4.13. From this figure it can be seen that all the responses in the three reactors

followed a similar pattern with the exception of reactor C between days 192-211. The concentrations in membrane C were lower than the other membranes until day 178, and then increased substantially for two sampling events then decreased to concentrations that were similar to the other two reactors. An ANOVA test with a significance level of 95% was conducted to compare the data sets and no significant difference was found as shown in appendix I. It was concluded that the temperature did not impact on biopolymers in the reactors in this phase.

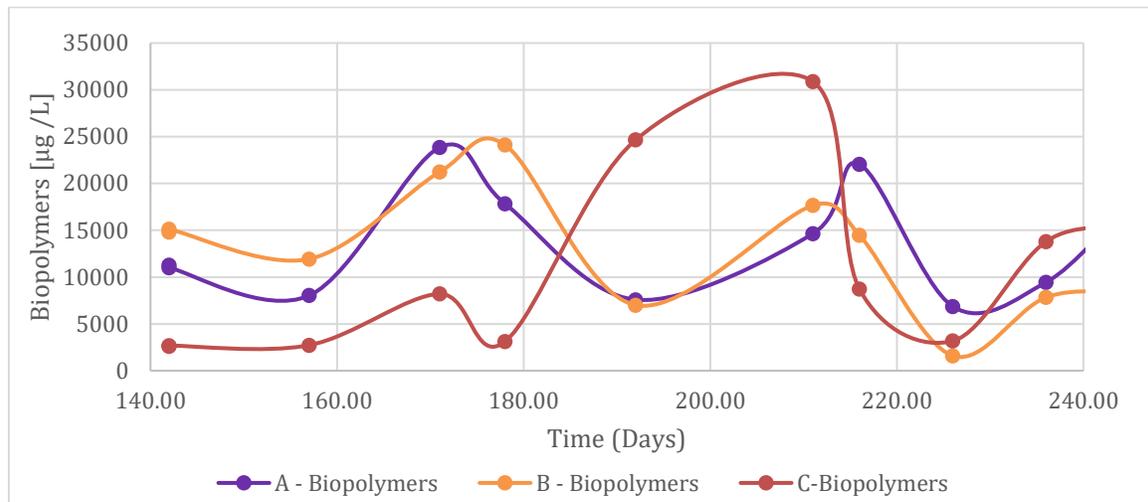


Figure 4. 13: Biopolymers concentrations for phase 1

The building blocks (BB) fraction was evaluated to assess whether they contribute to membrane fouling or pass through the system as suggested by Gibert et al, (2015). Figure 4.14 illustrates the concentration of BB during phase 1. It can be observed that from days 140 -178, reactor A had a large increase in BB while reactor B had a rise in BB levels on days 192 through to 211. Aside from these two increases, the fluctuations in the reactors were consistent. Similar to biopolymers, the concentrations in reactor C had the lowest concentrations with the exception of days 192 to 211, after which the concentrations in this reactor returned to the lowest values. An ANOVA test shown in Appendix I that was conducted at a confidence interval of 92% revealed that the three data sets were not significantly different. Hence, it was concluded that temperature did not significantly affect the BB response in the bioreactors.

In summary, all three NOM fractions showed no statistically significant difference among the three reactors. This suggests that they were not influenced by temperature within the tested range. The variation in temperatures was expected to affect the biological processes and the viscosity of the WAS. This suggests that their behavior could be attributed to fluctuations in the feed with minimal biological removal or generation. As such they would contribute to nbsCOD that has been previously described in the permeate. Overall, the lack of relationship between temperature and the NOM concentrations, suggests that the evaluated NOM fractions were not responsible for the increased fouling observed at psychrophilic temperatures which will be described subsequently.

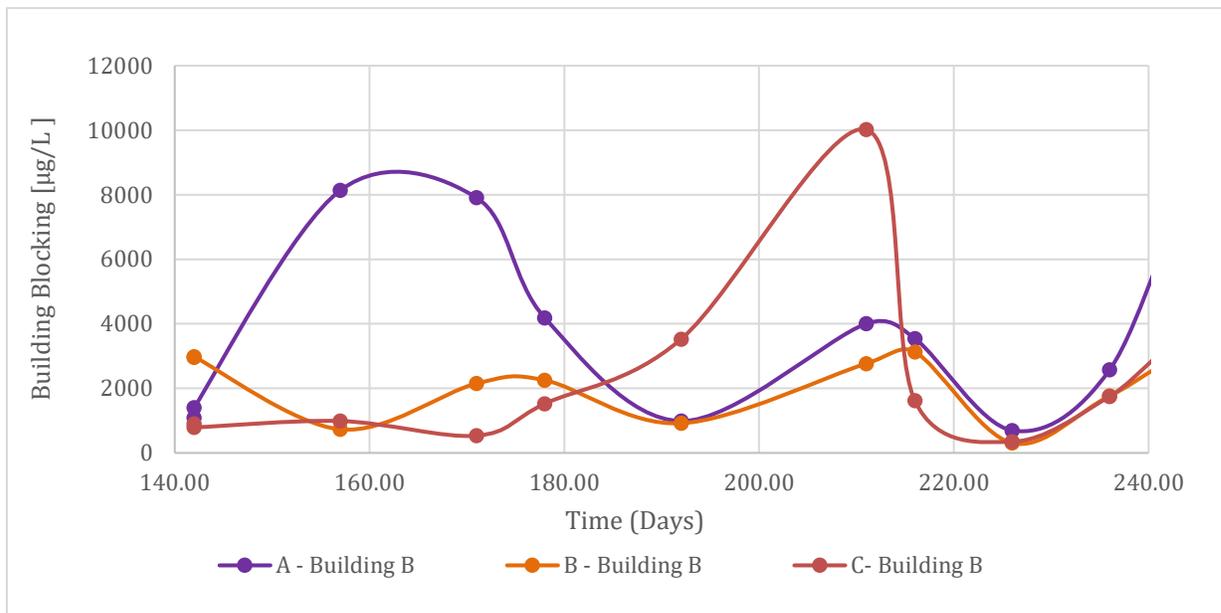


Figure 4. 14: Concentration of Building Blocks over phase 1

Cleaning frequency

The need for cleaning was employed as an indicator of the fouling propensity of the bioreactor contents. Three types of maintenance cleaning methods were implemented, as described in Section 3.2.3. The duration that each reactor operated before the need for a rinse clean was employed to characterize membrane performance. On average, reactors A and B operated for 3.13 and 3.28 days between rinses, respectively, whereas reactor C ran for 9.93 days with corresponding standard deviations of 1.04, 1.41 and 1.84. The histogram of values (Figure 4.15) reveals that reactors A and B had a normally distributed data set that was unimodal. Reactor C is bimodal so a conclusion cannot be drawn from the histogram. Kurtosis and skewness tests were applied to all three reactors as shown in Appendix Q

and these revealed that the data sets were normally distributed. The results indicated that the membrane fouling was more prominent at cooler temperatures as shown by the ANOVA and Tukey tests in Appendix Q. Since it was established that both the NOM fractions and TSS were not significantly different between operating at 10°C vs. 24°C, it was concluded that they did not affect fouling. It was concluded that SCOD and colloidal particles were indicative of the reduced cycle lengths at the cooler temperature.

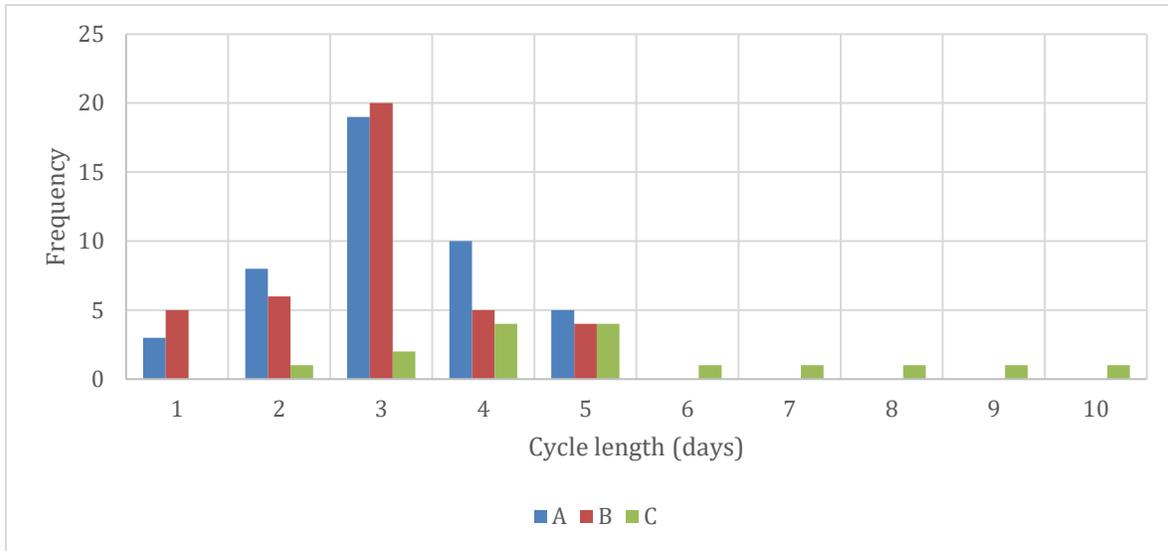


Figure 4. 15: Frequency distribution of cycle lengths of each reactor

4.2.3 Conclusions of Phase 1

The aim of phase 1 was to establish a baseline to contrast with phase 2 and to evaluate relationships with temperature. The parameters measured include, TCOD, SCOD, VSS, LC-OCD fractions and cleaning frequency. When tested for significance with regard to temperature, it was identified that at lower temperatures there was reduction in VSS concentration, and increase in COD levels in the permeate. This can be attributed to methanogens attaining low permeate COD concentrations by removing VFAs. Methanogen growth could be expected to be reduced at low temperatures. However, analysis also showed that there was a decrease in TCOD concentrations in the WAS as the temperature increased, which is indicative that less hydrolysis was occurring at 24°C. This was unexpected and it would be worth investigating further to attain an explanation.

The LC-OCD analysis showed no significant difference between the two temperatures but it can be used as the baseline for the LC-OCD analysis in phase 2. This suggested that NOM was not influenced by temperature, therefore unaffected by biological

process and thus must have entered the reactor through the feed. Monitoring the cleaning frequency showed that the reactor at 10°C ran for 50% shorter than the reactor at 24°C. Overall, reactors functioned better at warmer temperatures as expected based on the literature review investigated in Chapter 2.

4.3 Phase 2 of Experimental Analysis

This section presents the results from the second phase of the testing. In this phase, AnMBRs A, B, and C were set up in parallel at 10°C with 0.5, 1.0 and 2.0 g/L PAC added to each one respectively. The aim of this phase was to determine the effect PAC has at psychrophilic temperatures on the bioreactor and membrane performance. To evaluate the bioreactor performance, TCOD, SCOD, TSS and VSS in the WAS of each reactor and the TCOD of the permeate were measured. The COD required for sulfate reduction was calculated and methane generation was deduced through COD balance. The TMP, concentration of NOM, and cleaning frequencies were measured to evaluate the membrane performance.

4.3.1 Validate Phase 2 Data

As explained in section 4.2.4, it was deemed to be important to validate data and this was done for both phases using the checks described earlier in phase 1. The concentration of SCOD in the WAS should be greater than the concentration of the TCOD in the permeate, based on the pore size of the membrane (0.04 µm) and the filter paper used to measure the SCOD (1.5 µm). This was applied to each reactor separately. Further, it was expected that the TCOD of the WAS would be proportional to the VSS measurements since this stream mostly consists of suspended solids. A typical ratio for bacterial biomass is 1.42 g COD/g VSS. Thus, theoretical TCOD concentrations were calculated for the WAS from the measured VSS values and they were compared to the measured TCOD in the WAS. At the end of the analysis shown in Appendix J, it was determined that the data collected were valid and any anomalies that were present were eliminated.

4.3.2 Analysis and Discussion

4.3.2.1 Bioreactor

Initially, the bioreactor data was assessed to determine when the reactors had reached a dynamic steady state. Based on the data in Appendix B it was observed that there were fluctuations after the addition of PAC into the system especially in the initial 30 days. In order to incorporate a safety factor and to remain consistent with phase 1 it was deemed that steady state was achieved from day 90 (3 SRT cycles) onwards, thus marking the beginning of phase 2 on day 330.

WAS

TCOD is the summation of both the bCOD and the nbCOD, which in turn determines how the COD will be, distributed through the system. Hydrolysis will degrade the sbCOD portion of the bCOD at a slow rate whereas the rbCOD will be rapidly degraded initially. The nbsCOD portion was expected to accumulate in the effluent as it will not be biodegraded and pass through the membrane. This left the nbpCOD and the sbCOD, which were anticipated to contribute to the TCOD in the WAS. A high level of the latter would indicate that hydrolysis was not happening rapidly and thus resulting in an abundance of both colloidal and particulate sbCOD. It was expected that these constituents could increase both cake and pore fouling. The WAS was sampled bi-weekly and TCOD analysis was conducted, the results of which are presented in Figure 4.16. From this figure it can be seen that the concentration of COD fluctuated between 5,000 mg/L to 22,000 mg/L in all three reactors. However, it was unclear if the reactors differed in COD concentrations between each other. In addition, the PAC exerts a COD which was calculated and subtracted from the TCOD, so only WW COD fractions were considered. The means and standard deviations were calculated to facilitate a comparison as illustrated in Table 4.1. Table 4.1 indicates increased COD concentrations in reactors C and A as compared to B, while the standard deviations were similar. To determine if the differences were statistically significant, an ANOVA was conducted as shown in Appendix L, and it was concluded that the concentrations were not statistically significant. Hence, it was concluded that the sum of the nbpCOD and sbCOD components in the WAS were not altered due to the variation in PAC. However, it might be worth investigating the COD fractions individually as PAC could be acting as an adsorbent for nbpCOD or it could be enhancing biodegradation on the biofilm.

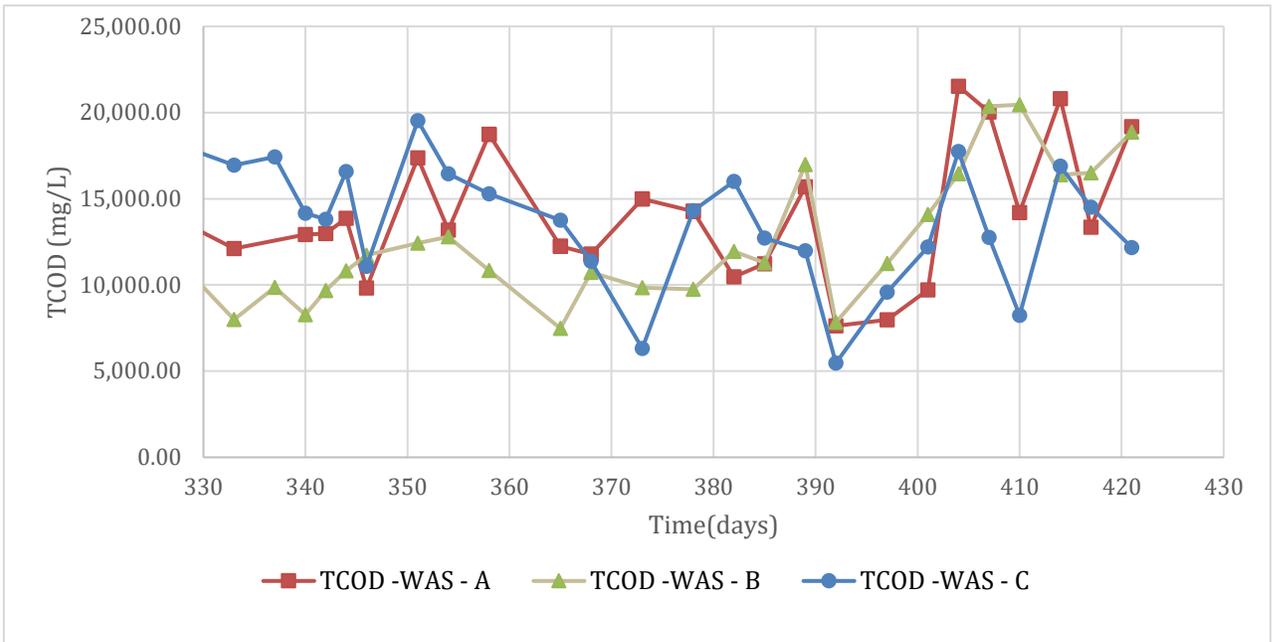


Figure 4.16: TCOD in the WAS for phase 2

Table 4. 1: TCOD Averages for Phase 2

TCOD	A	B	C
Average	14,072	12,586	13,493
SD	3,636	3,806	3,462

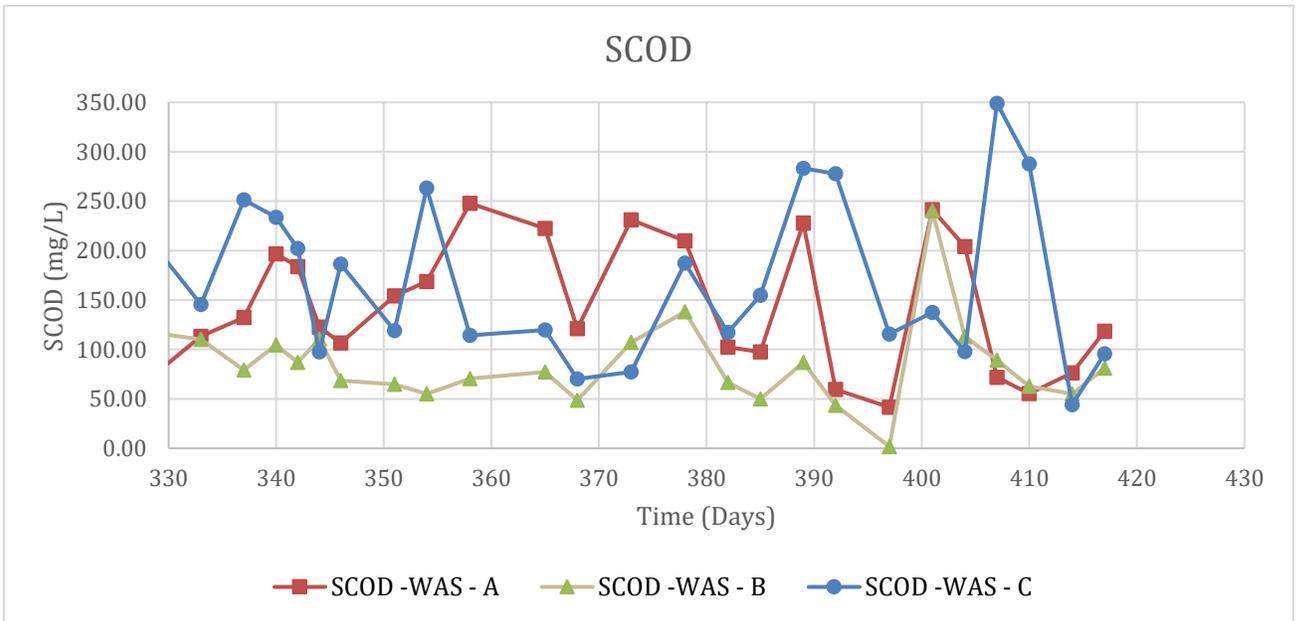


Figure 4.17: SCOD in WAS in all reactors for phase 2

Table 4. 2: SCOD in WAS for Phase 2

SCOD	A	B	C
Average	146.06	80.42	167.83
SD	65.02	25.10	82.75

SCOD represents the summation of nbsCOD, colloidal COD and rbCOD. It was expected that the rbCOD would rapidly degrade in the AnMBRs and hence was assumed to be negligible in the WAS. The fate of nbsCOD would depend on the particle size as particles smaller than 0.04 μm would filter into the permeate while larger particles could cause membrane fouling. The colloidal COD is also a possible foulant that can reduce membrane performance. Since both of these foulants could potentially be adsorbed by the PAC, they were evaluated in detail. The SCOD was measured in all WAS samples and the results are shown in Figure 4.17 The SCOD in the WAS fluctuated from 50 mg/L to 300 mg/L in the reactors over the course of testing. In addition, it can be seen that reactor B had the lowest concentration of SCOD throughout most of the phase. An ANOVA test was completed along with a Tukey test (see Appendix L) to assess whether the concentrations were significantly different. The testing revealed that the concentrations in Reactor B were significantly lower than the other two reactors. It was hypothesized that PAC would act as an adsorbent for nbsCOD and colloidal COD. Since the PAC is larger than the pores on the filter paper, PAC would be retained on the filter paper, thus the concentration of SCOD decreased in reactor B. In reactor A, the concentration of PAC may have been too low to cause a noticeable adsorption effect. In reactor C, the concentrations of SCOD were higher than in reactor B even though it was supplied a higher concentration of PAC. The cause of this behaviour requires further investigation but a possible explanation is proposed. It is hypothesized that PAC was scouring biofilm from the membrane in the reactors. However, it was also expected that biodegradation occurred in the biofilm. Hence, it is possible that in reactor C, due to the high concentration of PAC, the biofilm did not accumulate and thus less biodegradation occurred. This would explain the increased levels of SCOD present in the WAS of reactor C. Overall, adding a concentration of 1.0 g/L of PAC reduced SCOD levels in the WAS.

TSS in the bioreactors reflect the net impact of growth, endogenous decay and accumulation of feed particulates due to the difference between HRT and SRT. TSS is

comprised of VSS and Fixed Suspended Solids (FSS). The VSS is a reflection of the bacteria present in the reactor while the FSS is a representation of the inert nonvolatile particles. The TSS values are presented in Figure 4.18. In Figure 4.18, it can be seen that the TSS concentrations fluctuated between 5,000 to 35,000 mg/L throughout phase 2 of the experiment. An ANOVA test was executed as shown in Appendix L with a confidence level of 95%, to determine if either the VSS or FSS concentrations were significantly different between the three reactors. It was found that there was no difference in VSS concentrations but the FSS differed significantly thus, a Tukey test was executed. This identified that reactor C had a significantly higher FSS concentration than the other reactors. The elevated TSS was attributed to the presence of PAC that would contribute to the FSS component. When testing for FSS, the sample was placed in the oven at 550°C, so any organics would burn, leaving behind FSS. The decomposition of PAC occurs at approximately at 600 – 900°C (Bagreev et al., 2001) thus the PAC would not be burnt in the oven. Thus any PAC present would contribute to the FSS component. The VSS was considered to be representative of the biomass and the bacterial growth in the reactor and varying the concentration of PAC was found to have no significant impact on the VSS concentrations as shown in Appendix L. Thus, it was concluded that varying concentration of PAC did not have an influence on the bacterial growth. This indicates that PAC was not adsorbing organics and providing a surface area for biological growth. PAC was expected to adsorb foulants as mentioned earlier this could have occurred as the PAC used was too small. It could be useful to test varying AC sizes.

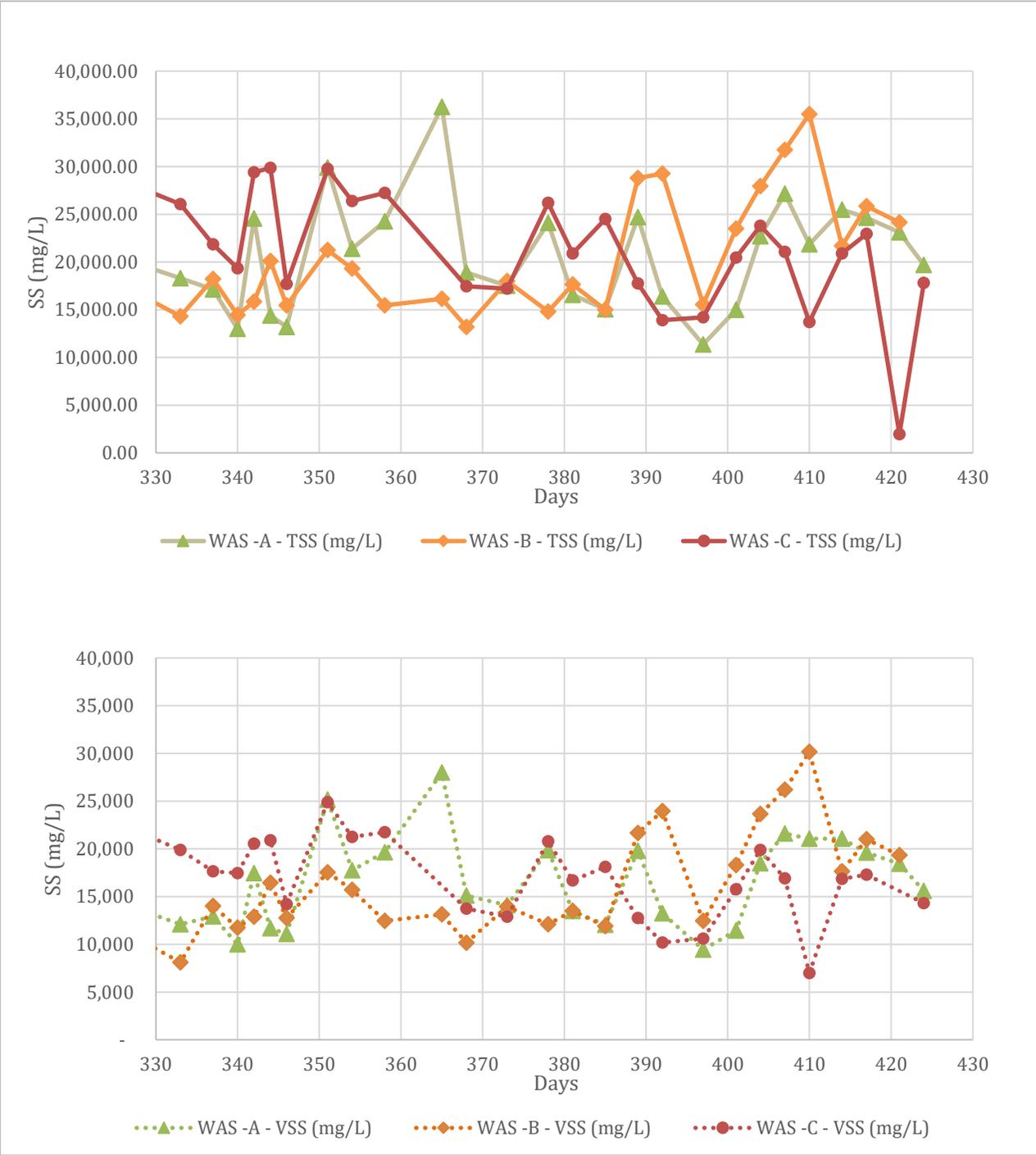


Figure 4.18: TSS and VSS in reactors during phase 2

Permeate

The permeate quality was assessed as a measure of system performance as it is a product of the reactors. All the nbsCOD in the feed and the products of acetogenesis would have contributed to the TCOD in the permeate. Hence, it provides a measure of the efficiency of the system as a whole and can be used to interpret how PAC was contributing to the functioning of the reactors. The permeate COD concentrations of all three reactors have been plotted in Figure 4.19 and the means and standard are presented in Table 4.3. The means were compared by ANOVA (Appendix M) where a significant difference was identified and thus a Tukey test was completed. It was determined that reactor B had a significantly higher quality of permeate as the COD levels were lower. This is indicative that PAC could be adsorbing nbsCOD. In addition it is the reactor with 1g/L PAC which had the highest quality of permeate which further supports the hypothesis previously discussed to explain the “dip” in the sCOD levels.

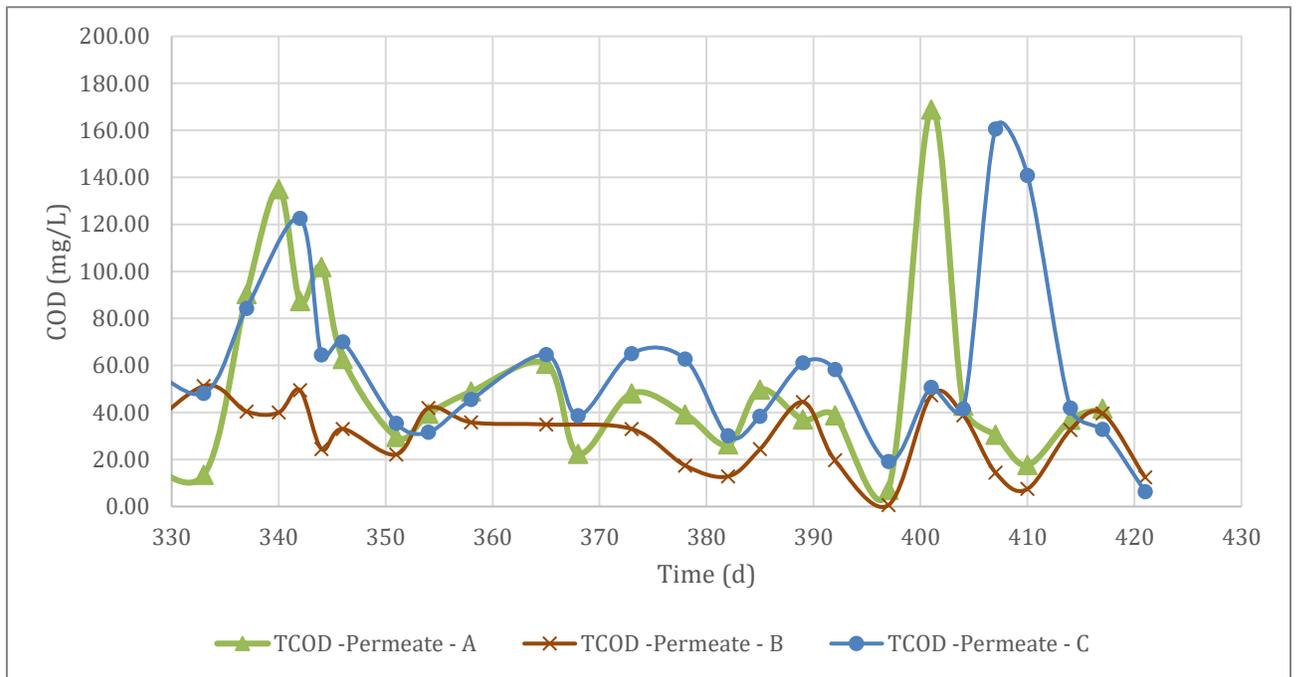


Figure 4.19: COD in permeate samples for reactors I phase 2

Table 4. 3: Average permeate TCOD for phase 2

	Average After PAC	SD
A	49.31	37.77
B	35.31	13.77
C	48.96	35.90

COD Balance

It was expected that the COD exits the reactors through 4 pathways; WAS, permeate, in the form of methane, or it could be used for sulfate reduction. Hence, in theory the summation of all four mass flows should be equal to the mass flow of COD in the feed based on the law of conservation. The COD used for sulfate reduction was calculated as shown in Appendix O. The COD in both the WAS and the permeate were measured and a cumulative analysis was used to convert the concentrations into a cumulative mass over the phase. In addition, the COD in the added PAC was accounted for using mass balance equations. The COD present as methane was calculated as the missing COD. Figure 4.20 summarizes the COD distribution throughout the reactors in phase 2.

It was noted that the COD associated with methane in Figure 4.20 appeared to be higher than expected. This was based upon the observation that, when converted to volume of biogas, the expected values were greater than 1L per day. This was in contrast with the lack of measurable gas phase methane production noted during the testing despite numerous attempts made to measure it. Hence, there is considerable uncertainty in the estimates of methane production. It was hypothesized that the COD balance was not fully closed and hence firm conclusions could not be drawn about the absolute values of methane generation. Rather, the values were used to make relative comparisons between reactors.

The results were analyzed to compare the mechanisms that were active in each reactor relative to one another. Statistical analyses were applied for each pathway individually as discussed earlier. From Figure 4.20, it can be seen that reactors A and C had a higher fraction of the COD in the WAS which suggested that more hydrolysis was occurring in reactor B in comparison to the other two. This was not an expected set of results as the concentration of PAC was increasing from reactor A to C. Similarly, reactor B had the cleanest permeate (4.24%) and the highest production of methane (63.58%) in comparison. A potential hypothesis can be drawn based on the two main functions of PAC. PAC is extremely adsorptive and can adsorb soluble COD, and it also has scouring properties which can remove biofilm formed on the membrane. However, if significant biodegradation occurs on the biofilm and it is removed, then less hydrolysis will occur. Hence, in reactor C, the concentration of PAC was higher so it was scouring more biofilm from the membrane and thus less biodegradation occurred. This explains why Reactor C had a higher concentration of COD in the WAS. Overall, it would appear that at a PAC concentration of 1

g/L, the PAC was adsorbing COD fractions while forming a substantial layer of biofilm on the membrane which enabled biodegradation.

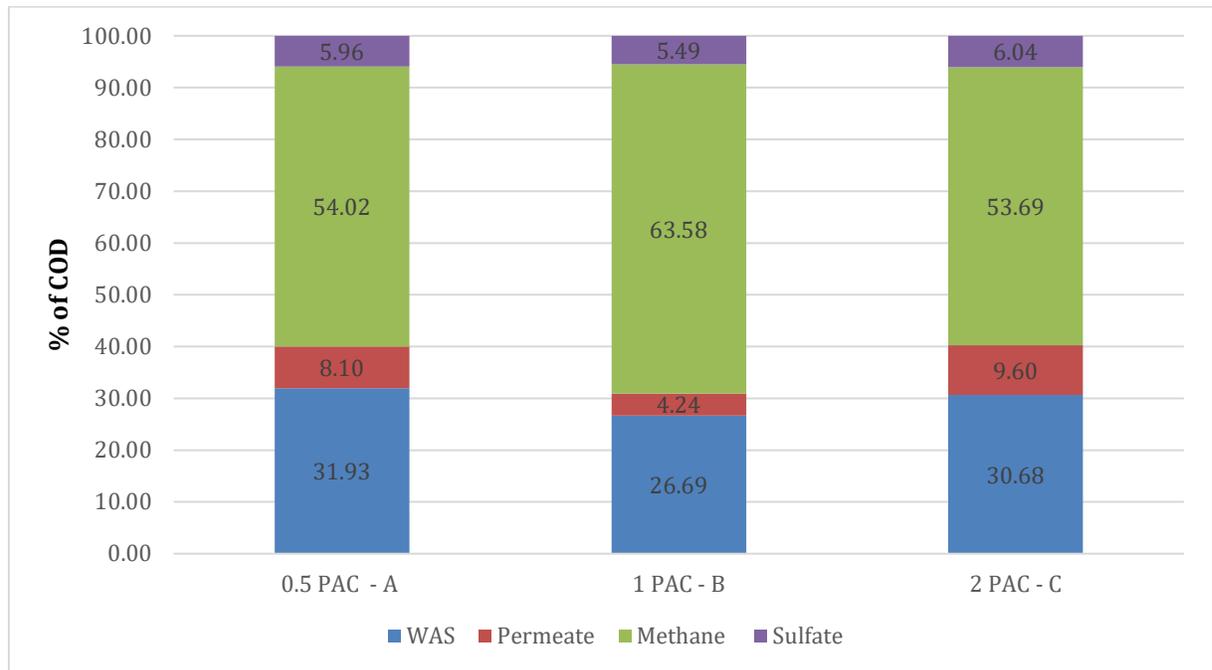


Figure 4.20: COD distribution in all reactors in phase 2

4.3.2.2 Membrane

LC-OCD

LC-OCD was used to measure fractional concentrations of NOM constituents in samples generated in this study that were hypothesized to be indicators of potential fouling. As previously stated, it was anticipated that the NOM fractions could either be generated, degraded or remain unaffected in the AnMBR. In section 4.2.3.2, it was established that the responses of the NOM fractions in the AnMBRs were unaffected by temperature, thus it was concluded that they were not generated or degraded. Instead, they were thought to contribute to the nbsCOD in the permeate. However, it was expected that PAC addition could result in NOM adsorption. Hence, the fractions were characterized in this phase to explore the combined effects of adsorption and biodegradation.

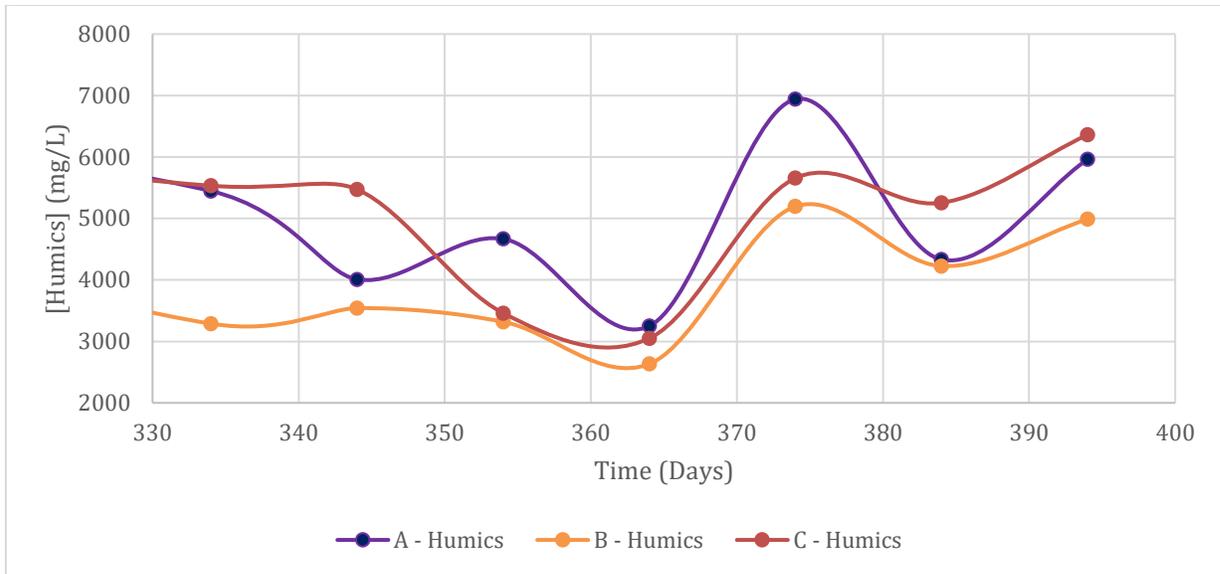


Figure 4.21: Humics concentrations in the WAS during Phase 2

Humics

Some studies (Thekkedath, 2010) reported that humics were mainly governed by cake formation mechanism hence their fate was evaluated in this study as one part of an overall assessment of the potential of PAC to reduce membrane fouling. The concentration of humics in the WAS fluctuated with time throughout Phase 2 as can be observed in Figure 4.21. It was assumed that external factors such as variation in the feed composition caused the large fluctuations in the humics concentration as the fluctuations were observed in all the reactors. An ANOVA was applied to determine if the data was significantly different and the findings have been summarized in Appendix P. It was determined there was no significant difference between the three reactors. It was expected that adding PAC would increase adsorption of humics but as there was large variation in the data, the difference in humics levels were not significantly different in the reactors. Hence, adding PAC did not appear to have an impact on the response of humics in the AnMBRs in this study.

Biopolymers

Biopolymers consist of proteins and carbohydrates and are slightly larger than humics, thus more suited for adsorption and can contribute to the formation of biofilms. (Croft, 2012) Figure 4.22 illustrates the biopolymer concentrations in the WAS over the course of testing. Reactor B had a lower concentration of biopolymers for a majority of the time, especially after day 290. An ANOVA at a significance level of 95% was used to assess whether there was a significant difference between the reactors. The ANOVA revealed there

was a difference and a Tukey test revealed that Reactor B had significantly lower concentrations (Appendix P). The results suggest that adding PAC at a concentration of 1 g/L reduced the concentration of biopolymers in the reactors, which was hypothesized to reduce fouling. This was consistent with the results for SCOD in the WAS, where reactor B had a lower concentration of SCOD which lead to the conclusion that PAC was adsorbing the nbsCOD. The LC-OCD results suggest that similarly, PAC was adsorbing biopolymers. By contrast, adding more PAC (2 g/L) in reactor C did not decrease the biopolymer concentration. As hypothesized earlier, this could be because the increased concentration of PAC was reducing biofilm formation in reactor C. Hence, the biopolymers enmeshed in the biofilm were being redistributed through the WAS. Thus, adding 1 g/L of PAC can significantly reduce the concentration of biopolymers in the WAS, which in turn could reduce membrane fouling.

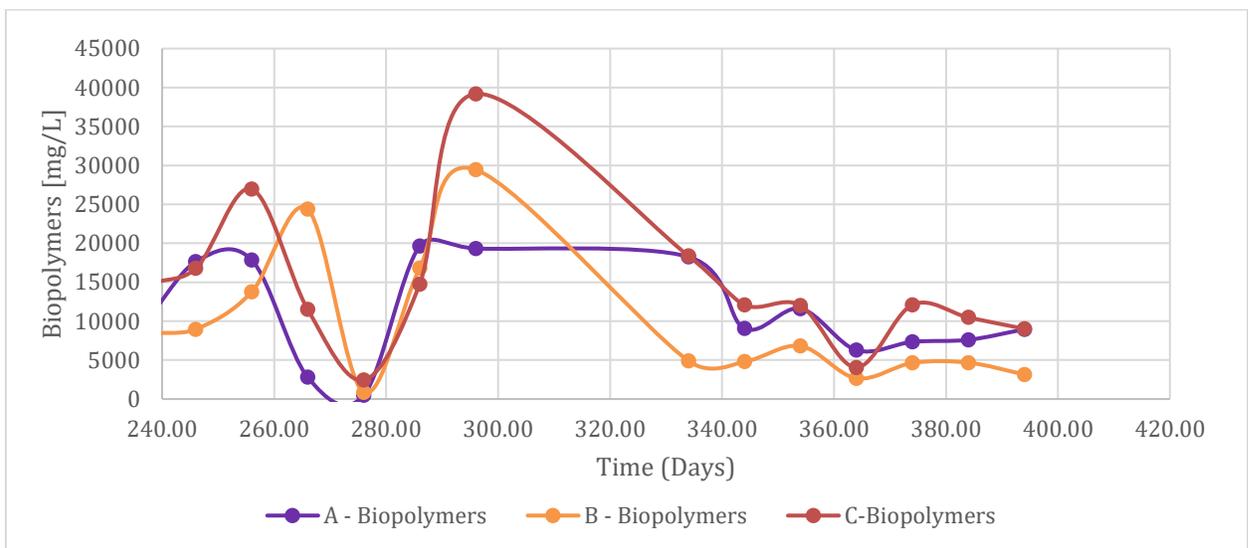


Figure 4.22: Biopolymer concentration in Phase 2

Building Blocks

Building Blocks (BB) are weathering and oxidation products of HS; they cannot be removed during flocculation processes. Although it is rare building blocks foul membranes, but it was worth investigating to observe potential fouling and the effect PAC can have on it. The concentrations of BB in the WAS of each reactor are shown in Figure 4.23. An ANOVA was done at a significance level of 95% and it was determined that there was no significant difference between the three reactors, hence there was no dependence on the concentration of PAC added to the reactor. Thus, it showed that the BB was not being adsorbed onto the

PAC, and in the analysis of Phase 1 it was determined that the NOMs were not generated or biodegraded in the reactor. As there was no correlation between BB concentration and the addition of PAC, it was assumed that BB continues to enter the reactor through the feed and is unaffected by PAC as in Phase 1.

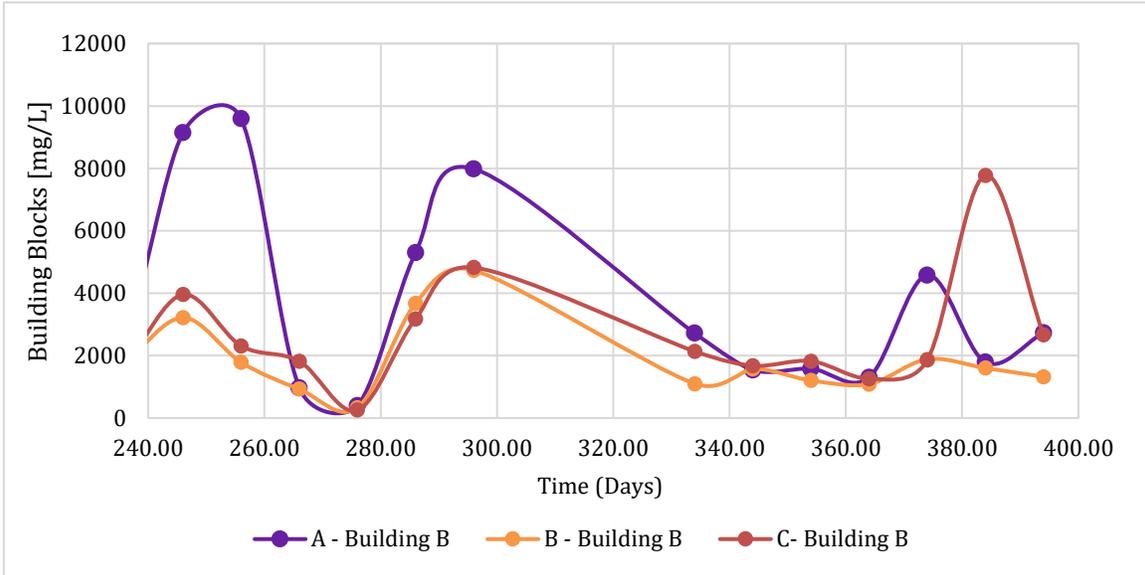


Figure 4. 23: Building Blocks concentration in all reactors in phase 2

Cleaning Frequency

The need for rinse cleans was employed as an indicator of the fouling tendency of the reactors. The duration that the reactors operated before a rinse clean was needed was used to characterize membrane performance. The full set of results are displayed in Appendix B and a frequency plot is shown in Figure 4.24. On average, reactors A, B and C operated for 5.86, 6.43 and 6.84 days between cleans with standard deviations of 1.50, 1.05 and 1.23 respectively. All three data tests were unimodal and to test if the datasets were normally distributed they were subjected to skewness and Kurtosis tests as shown in Appendix Q. The tests indicated that the datasets were normally distributed. The mode for each reactor was 7 and when an ANOVA was conducted at a confidence interval of 95% it was found that there was not a significant difference between the three reactors. This shows that varying the concentration of PAC had no effect on the cleaning frequency. This was unexpected as adding a higher concentration of PAC should in theory act as a stronger abrasive material. However, this could be because the membrane was cleaned before it was

completely fouled. Hence, it was hypothesized that the TMP would be a more accurate representation of membrane performance.

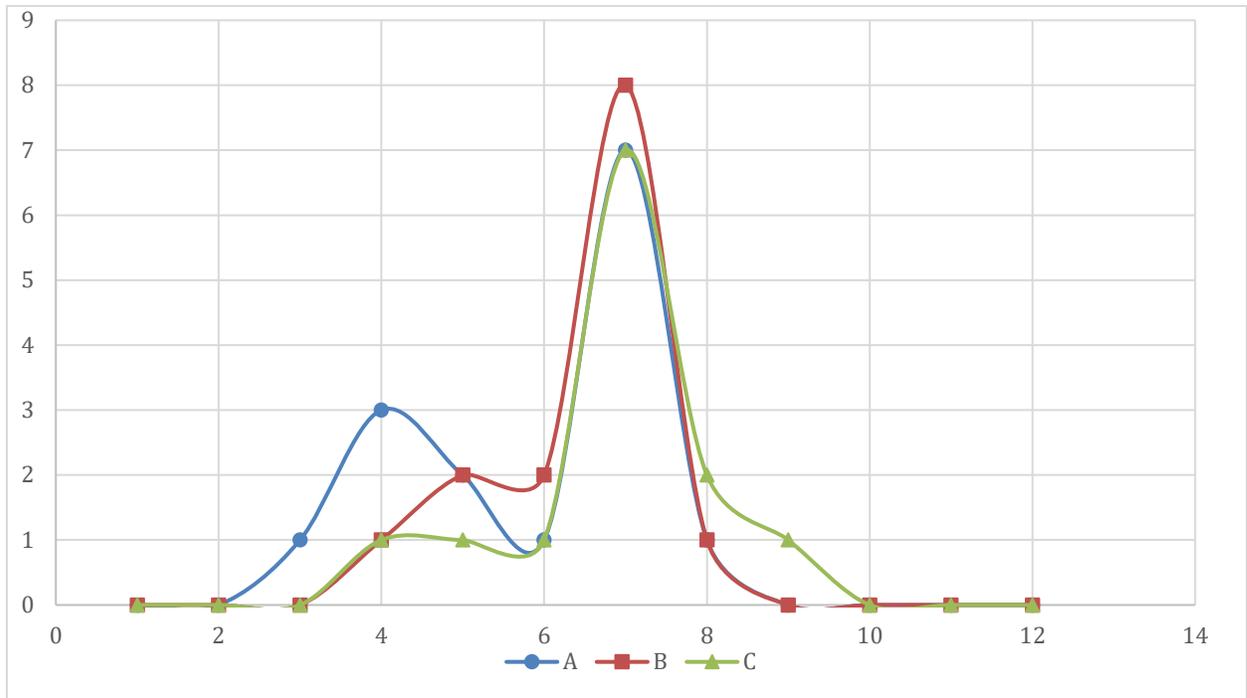


Figure 4. 24: Frequency plot for cycle length

Trans membrane pressure

In constant flux operation, a sharp increase in TMP was considered to be indicative of severe membrane fouling. This sudden TMP increase has been called a “TMP jump” (Chen et al., 2006; Zang et al., 2006). TMP jump has been described as a three-stage process Stage 1: an initial “conditioning” fouling, which is caused by initial pore blocking and solutes adsorption. Stage 2: linear or weakly exponential gradual rise in TMP due to biofilm formation and further membrane pore blocking. Stage 3: a sudden rapid increase in the rate of TMP rise ($dTMP/dt$). Stage 3 is thought to be the consequence of severe membrane fouling, and is believed to be due to successive closure of pores and changes to the local flux resulting from fouling, which causes local fluxes to exceed the critical value. Typically at this stage, a rinse clean was conducted, but often the cleaning was done before this occurred. In order to analyze the TMP, 10 cycles that reached up to stage 3 were evaluated to determine the cycle length for each reactor. Figure 4.25 shows a snapshot of a cycle for each reactor, and the remaining data were repeats of the similar cycles. When the pump was at rest, the TMP was at 4.9 Hg and at vacuum it dropped to 0. The small fluctuations between 4.9 and

4.4 Hg resulted from the rest and operational periods of the membrane as the permeate pump was operated for eight minutes and allowed to rest for two minute. From the three graphs it can be observed that there was a slight decrease in TMP over time and the three stages described can be clearly seen. This indicates that the membranes behaved in the manner described by Chen et al., (2006) and Zang et al., (2006).

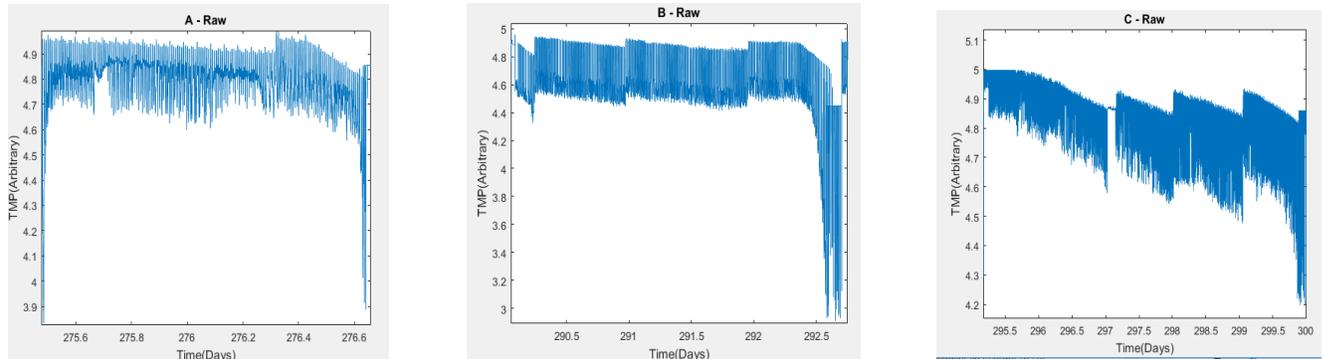


Figure 4. 25: TMP for ABC for a cycle

	Average Cycle Length	SD
A	5.94	0.79
B	7.06	0.68
C	8.24	0.78

Table 4. 4: Average cycle length for Phase 2

The TMP dataset was densely populated over the course of the experiment, and to analyze it simultaneously was difficult and inefficient as there were several fluctuations within day-to-day variations. Hence, 10 cycles that reached up to stage 3 were evaluated to determine the cycle length for each reactor. The averages are summarized in Table 4.4. It was found that AnMBRS A, B and C had average cycle lengths of 5.9, 7.1 and 8.2 days respectively. In comparison, the average cycle lengths from the cleaning frequency were 5.86, 6.43 and 6.84 days for A, B and C respectively. The cycle lengths depicted from cleaning frequencies were lower as in several cases the AnMBRs were cleaned before the reactor reached stage three whereas when analyzing TMP, 10 cycles that went through all three stages were selected. As shown in Appendix V, an ANOVA test at 95% significance level was conducted and a Tukey test was executed as a follow up and it was established that all three reactors' TMP cycles were significantly different. Hence, the results indicate that adding PAC can help with the scouring ability of the membrane and can thus extend the duration before the membrane needs to be cleaned.

4.3.3 Conclusion of Phase 2

The aim of Phase 2 was to determine the effect of three different concentrations of PAC on the reactors. When analyzing membrane performance based on the TMP and cleaning frequency, it was found that as the concentration of PAC was increased, the cycle lengths also increased, suggesting that PAC was acting as an abrasive. It was also noted that TMP was a more accurate method of measuring membrane fouling as each cycle is visible and an accurate cycle length can be established, especially if the system was cleaned in-between cycles. It was also found that Reactor B had a significant reduction in biopolymers and SCODs in the WAS. This was indicative of PAC acting as an adsorbent of these constituents in this reactor. In addition, it was clear that Reactor B had a cleaner permeate, suggesting that PAC could have also been adsorbing a portion of the nbsCOD or that more constituents were getting converted into methane. The fact these results were not observed in Reactor C suggested that the biodegradation was mainly occurring at the biofilm level and that since scouring increased with the concentration of PAC, biofilm was not accumulating in Reactor C. Overall, it was concluded that adding PAC at appropriate concentrations can aid in optimizing AnMBR performance.

5.0 Conclusions and Recommendations

5.1 Conclusions

This research was conducted to explore the effect that powdered activated carbon (PAC) has on the performance of AnMBRs in psychrotrophic conditions. The experiment was conducted in two stages, stage 1 investigated the effect of psychrotrophic conditions and stage 2 investigated the effect of PAC at 0.5, 1.0 and 2.0 g/L. In both stages the bioreactor and membrane performance were monitored and evaluated. The main conclusions from each stage are summarized below:

Stage 1

- There was no statistical difference in TSS in all three reactors, whereas the reactor at 24°C had significant higher VSS concentrations than the reactors at 10°C, this was anticipated due to the enhanced growth of the bacteria at higher temperatures.

- Analysis of membrane permeate quality demonstrated that the reactor at higher temperature (24°C) had a significantly lower concentration of COD, indicating that permeate quality decreased when the temperature dropped. This finding was consistent with pre-established model that methanogens were responsible for attaining low permeate COD concentrations by removing VFAs. Methanogen growth can be expected to be reduced at low temperatures.
- The concentrations of three NOM fractions (humic acids, building blocks and biopolymers) were measured in all three reactors, but none were significantly different from the others. This suggested that their removal was not influenced by temperature. This could be attributable to fluctuations in the feed with minimal biological removal or generation thus NOMs were not responsible for the increased membrane fouling observed at psychrophilic temperatures.
- Based on the cleaning frequency of the membranes, fouling was more prominent at the cooler temperature. The cycle lengths decreased from 9 days at 24°C to 3 days at 10°C.

Stage 2

- There was no significant difference in TCOD levels among the three PAC concentrations thus it was concluded that the sum of the nbpCOD and sbCOD components in the WAS was not altered due to the variation in PAC concentration. This suggests that either that PAC has no effect on both of them or that PAC could be affecting nbpCOD and sbCOD differently thus the summation of both shows effect.
- The reactor with the highest PAC concentration (2.0 g/L) had a significantly higher FSS concentration, whereas the VSS was not significantly impacted by PAC concentration. The elevated TSS is attributed to the presence of PAC that would contribute to the FSS component during testing. The lack of impact of PAC

concentration on VSS levels is an indication that PAC was not providing sufficient area for biological growth.

- The reactor with 1.0 g/L of PAC had a significantly lower level of SCOD in the WAS and TCOD in the permeate, resulting in a higher quality permeate. This was unexpected but is explained by two properties of PAC. PAC is acting as an adsorbent (nbsCOD & colloidal COD) and as a scouring agent. At the lowest concentration of 0.5 g/L the concentration of PAC may have been too low to cause a noticeable adsorption effect. At the highest level of 2.0 g/L the PAC was likely adsorbing but in addition causing significant scouring. Though the scouring improved membrane function, it removed the biofilm and thus biodegradation that occurred on it resulting in a lower quality of permeate and higher SCOD levels.
- The only NOM to be impacted by PAC concentration was the biopolymers. The reactor with 1.0 g/L PAC had significantly lower levels than the reactors with PAC concentrations of 0.5 and 2.0 g/L. This could be because the increased concentration of PAC was reducing biofilm formation in the reactor with 2.0 g/L PAC. Hence, the bio-polymers enmeshed in the biofilm were being redistributed through the WAS. Thus, adding 1 g/L of PAC significantly reduced the concentration of biopolymers in the WAS, which in turn could reduce membrane fouling.
- The cycle lengths increased significantly with the increase in PAC concentration from cycle lengths of 5.9, 7.1 and 8.2 days with PAC concentrations of 0.5, 1.0 and 2.0g/L days respectively. This is indicative that adding PAC can assist with the scouring of the membrane and thus extend the duration before the membrane needs to be cleaned.

Overall membrane fouling increased at psychrotrophic temperatures and bioreactor performance decreased. This can be mitigated to a degree with the addition of PAC. However, adding too much PAC can reduce the biodegradation on the biofilm hence it is important to add an optimum concentration of PAC to the WAS.

5.2 Recommendations

The results of this study suggested PAC addition could reduce membrane fouling and at specific concentrations improve bioreactor performance. Based on the findings in this current research some additional work is suggested below.

- If similar test was conducted, it would be ideal to measure methane generation so that the COD mass balances can be closed more reliably.
- Conduct more experiments at varying levels of PAC to determine the relationship between bioreactor performance and PAC concentrations. This can be used to identify the optimum PAC concentration.
- Complete a pilot scale study in order to determine if AnMBRs can be scaled up and whether similar results will occur.
- Setup a software simulation that can be used to incorporate different concentration of PAC, to view the effect it has on WAS, permeate and methane productions.

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Appendixes

A - Feed Significance test.....	2
B - Raw Data.....	4
C - WAS Cumulative Data for Phase 1.....	9
C1 - Cumulative TCOD Data.....	9
D - TSS VSS for Phase 1	12
E - Permeate for Phase 1.....	13
F - Sulphate reduction calculations	15
G - COD Balance for Phase 1	16
H - LMW form LCOCD for Phase 1	17
I - ANOVA for LCOCDs for Phase 1	18
J - Validations of Phase 1 and Phase 2 data.....	21
K - TMP.....	32
L - WAS Calculations for Phase 2	34
M - Permeate Calculations for Phase 2	36
N - Effect of PAC on % removal.....	38
O - COD Balance for Phase 2	38
P - LCOCD Calculations for Phase 2	40
Q -Cleaning frequency	44
Q1 - Significant difference in Cleaning frequency	44
R- Between phases comparison.....	45
R1 - Percentage Removal.....	45
R2 - Hypothesis testing.....	46
S - TCOD WAS Phase 1.....	49
T - LCOCD t -Test	51
T1 - Humics.....	51
T2 - Biopolymers	52
T3 - Building Block.....	53
U - T- test for Cleaning frequency.....	53
V- TMP Phase 2	54
W- Apparatus	55

A - Feed Significance test

A1 - Difference between Phases

Based on the table shown above I need to conduct the independent T-test to determine if the means of both Phase 1 and Phase 2 are significantly different.

	Phase 1 -AB	Phase 2 - AB
Mean	739.19	510.81
Variance	51804.76	27074.39
Observations	31.00	10.00
Hypothesized Mean Difference	0.00	
df	21.00	
t Stat	3.45	
P(T<=t) one-tail	0.00	
t Critical one-tail	1.72	
P(T<=t) two-tail	0.00	
t Critical two-tail	2.08	
There is a difference P<0.05		

	Phase 1 -C	Phase 2 - C
Mean	669.18	463.13
Variance	74683.22	24755.23
Observations	41.00	10.00
Hypothesized Mean Difference	0.00	
df	24.00	
t Stat	3.14	
P(T<=t) one-tail	0.00	
t Critical one-tail	1.71	
P(T<=t) two-tail	0.00	
t Critical two-tail	2.06	
There is a difference P<0.05		

This shows that there is a statistical difference in the Feed between phase 1 and 2 this can be explained by seasonal changes

A2 - Difference between AB and C

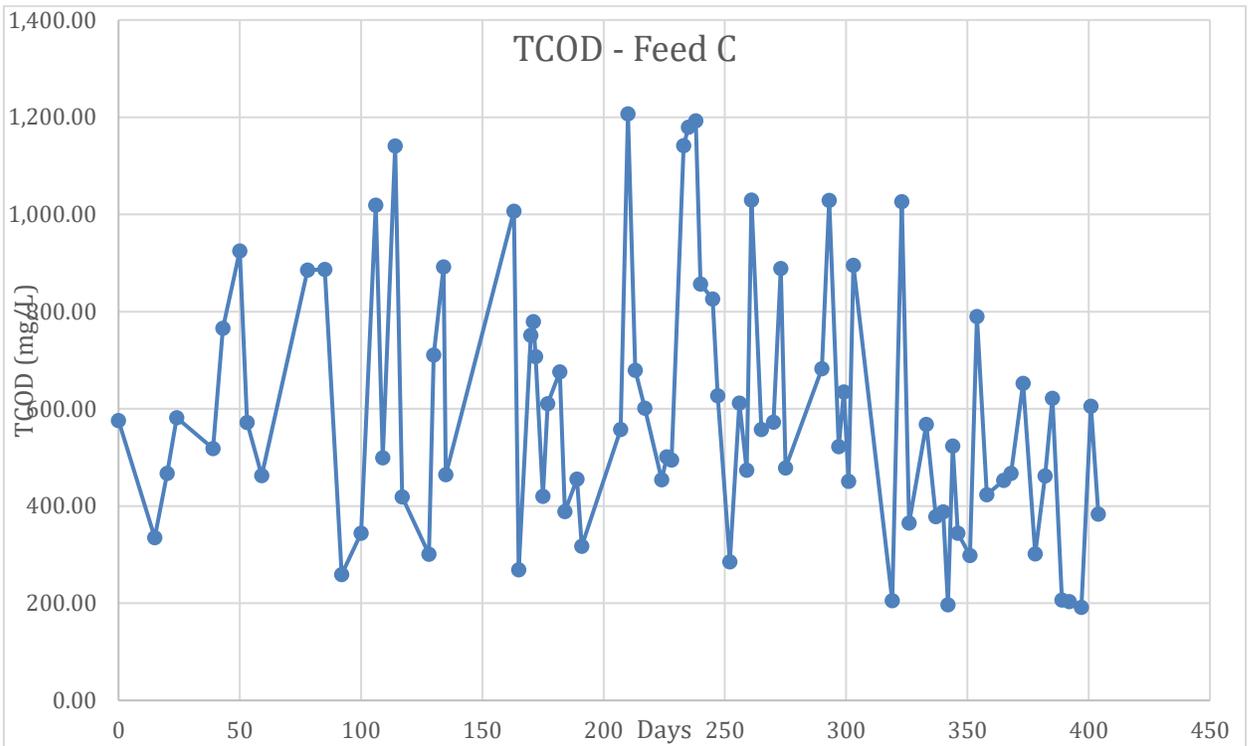
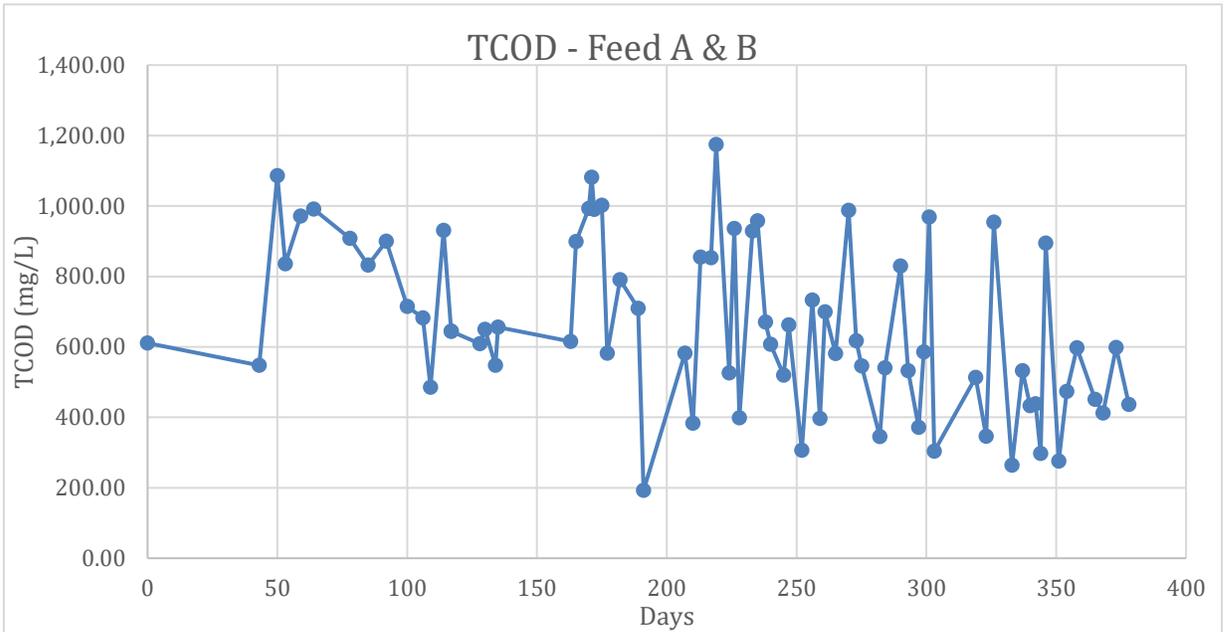
In order to determine if there is a significant difference between the three reactors, the T-test can be implemented and it has been summarized in the tables below. In theory, there should not be any difference between both the feed streams as the main sample is collected from the same pipeline. It is just stored in two different main containers. To determine this a hypothesis test can be executed.

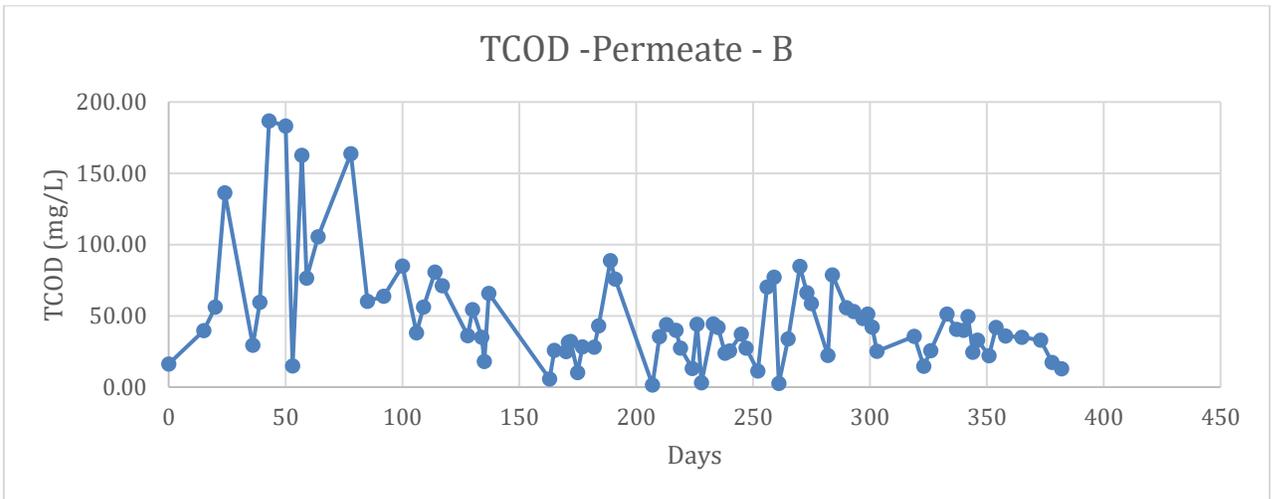
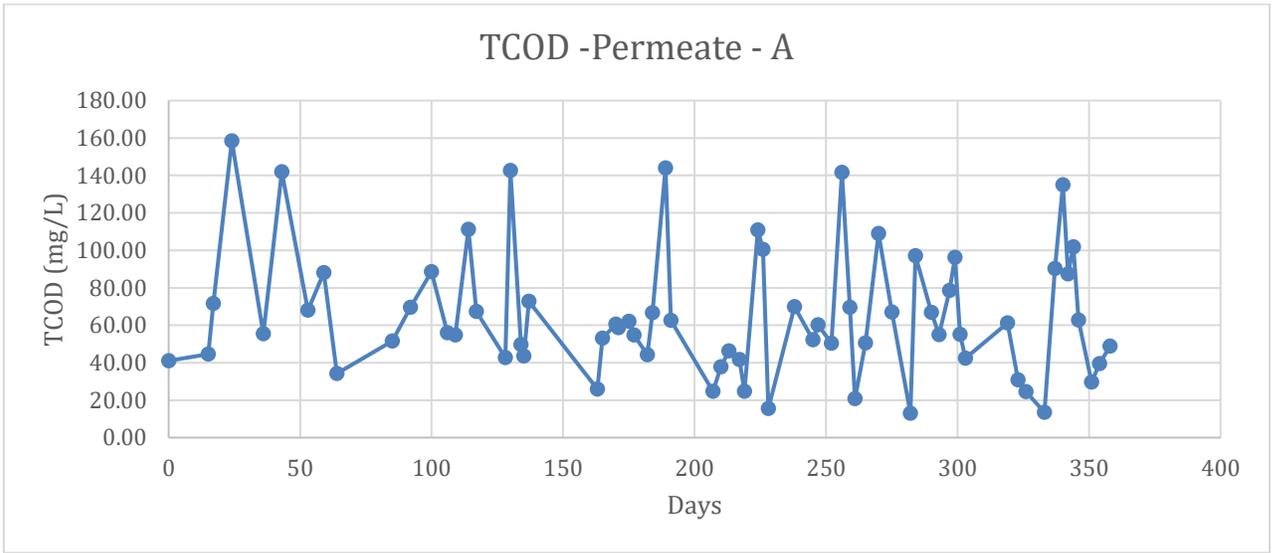
	Phase 1 - AB	Phase 1 - C
Mean	739.19	669.18
Variance	51804.76	74683.22
Observations	31.00	41.00
Hypothesized Mean Difference	0.00	
df	69.00	
t Stat	1.18	
P(T<=t) one-tail	0.12	
t Critical one-tail	1.67	
P(T<=t) two-tail	0.24	
t Critical two-tail	1.99	
There is a NO difference $P > 0.05$		

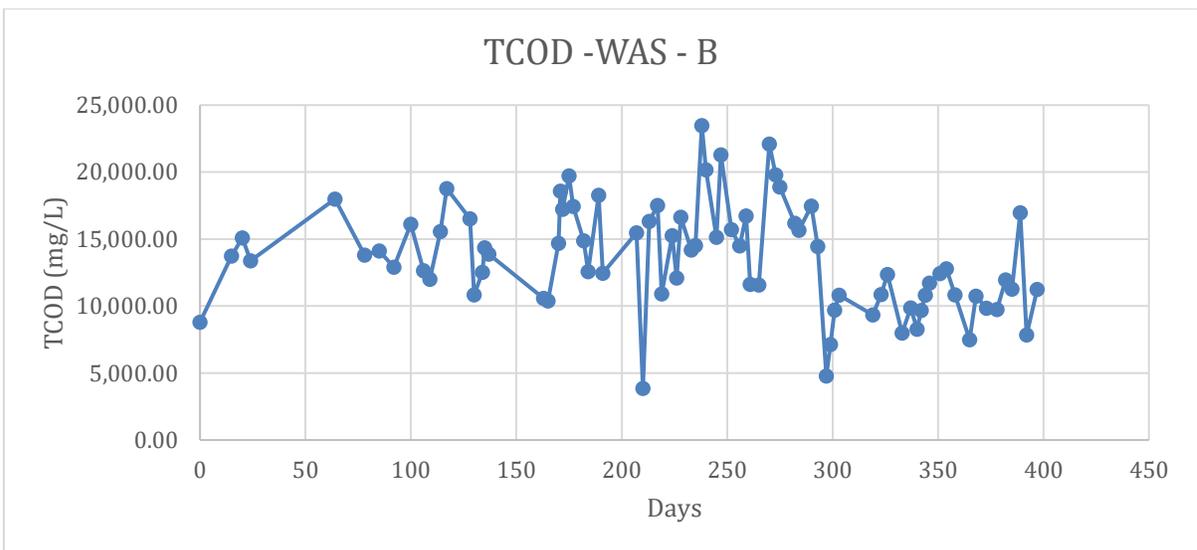
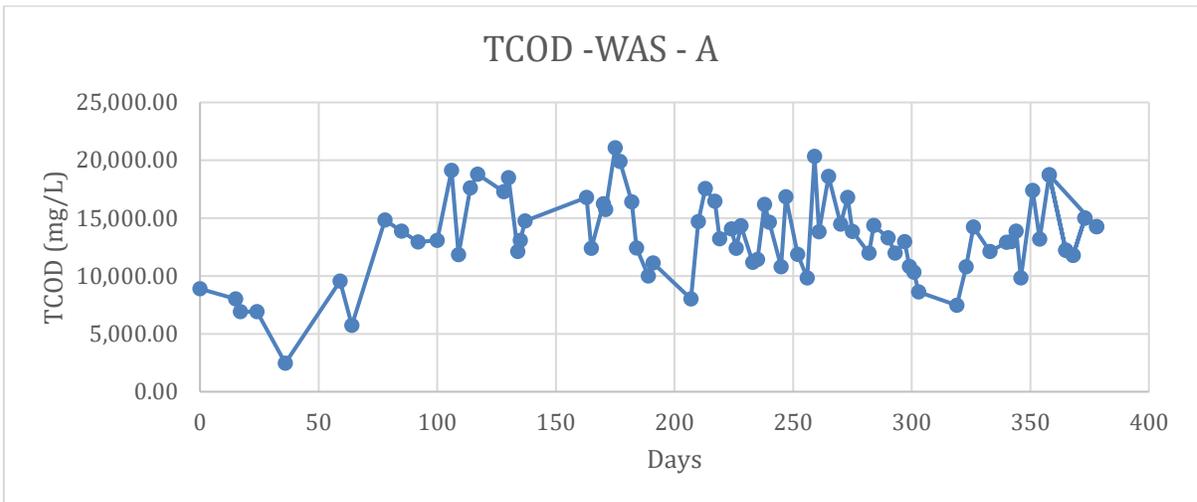
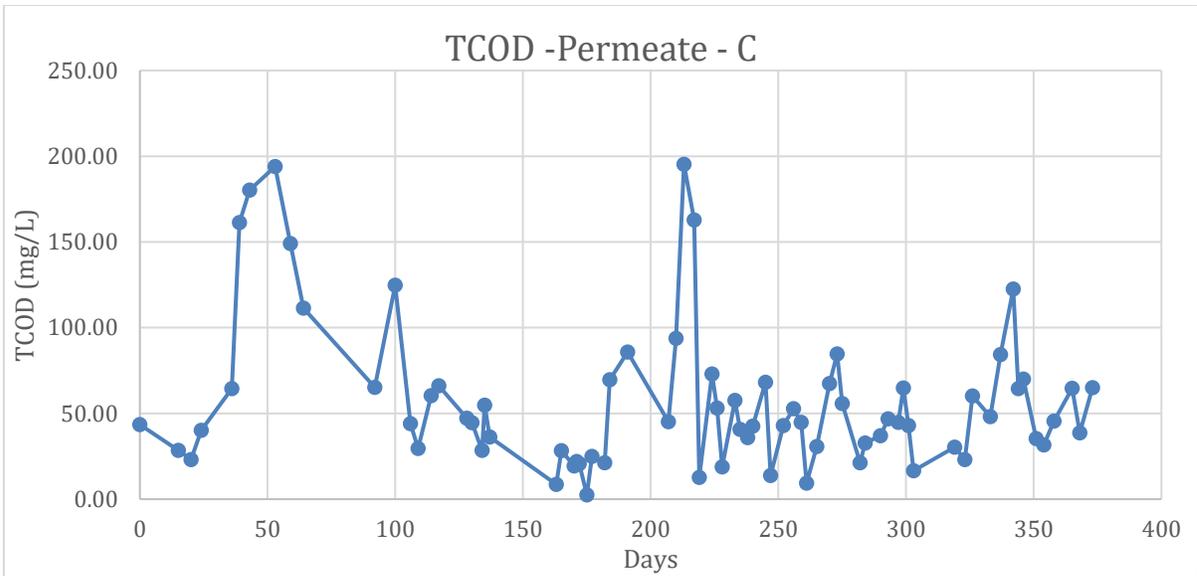
	Phase 2 - AB	Phase 2 - C
Mean	510.81	463.13
Variance	27074.39	24755.23
Observations	10.00	10.00
Hypothesized Mean Difference	0.00	
df	17.00	
t Stat	0.66	
P(T<=t) one-tail	0.26	
t Critical one-tail	1.74	
P(T<=t) two-tail	0.52	
t Critical two-tail	2.11	
There is a NO difference $P > 0.05$		

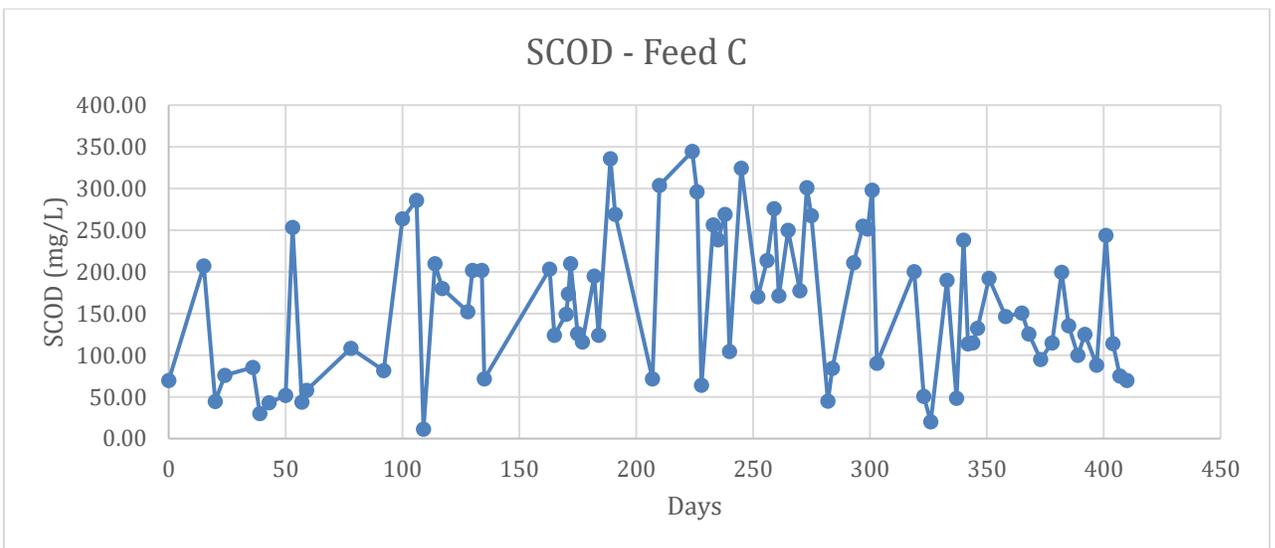
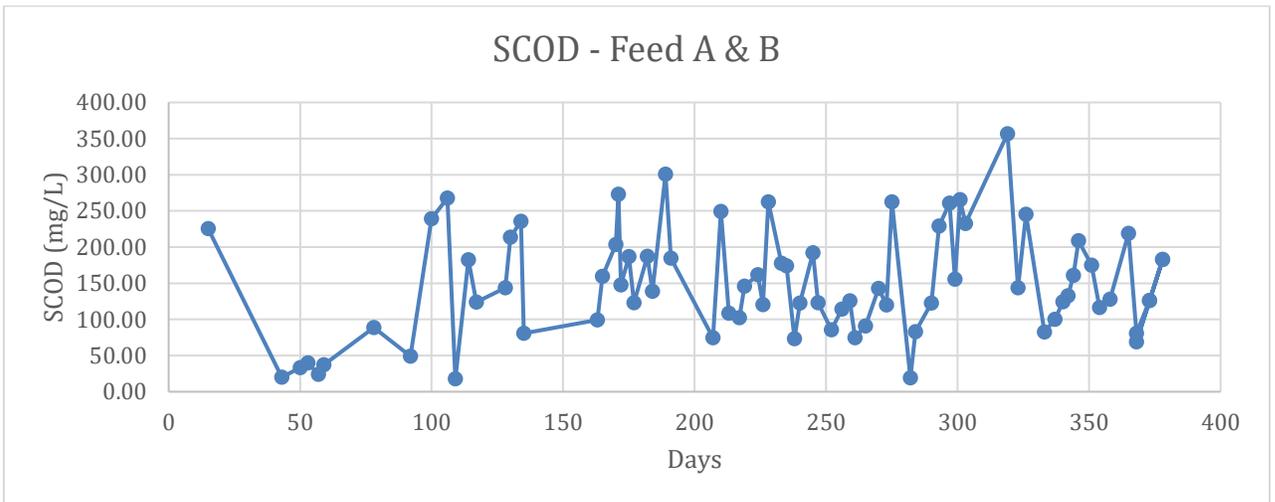
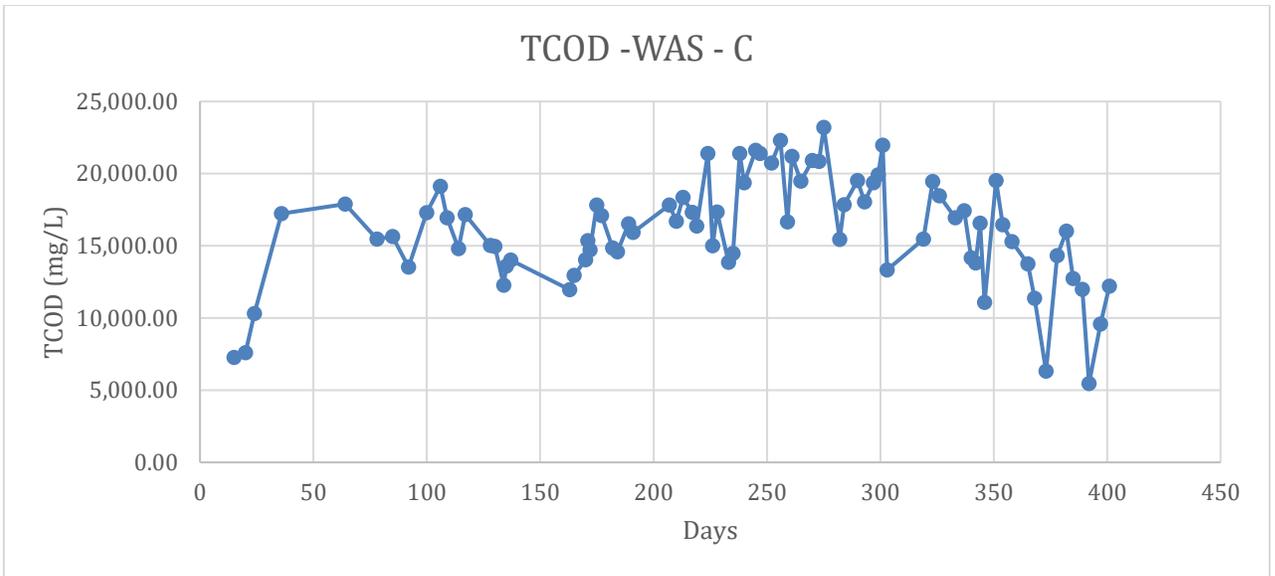
They both show that there is no significant difference between the three reactors.

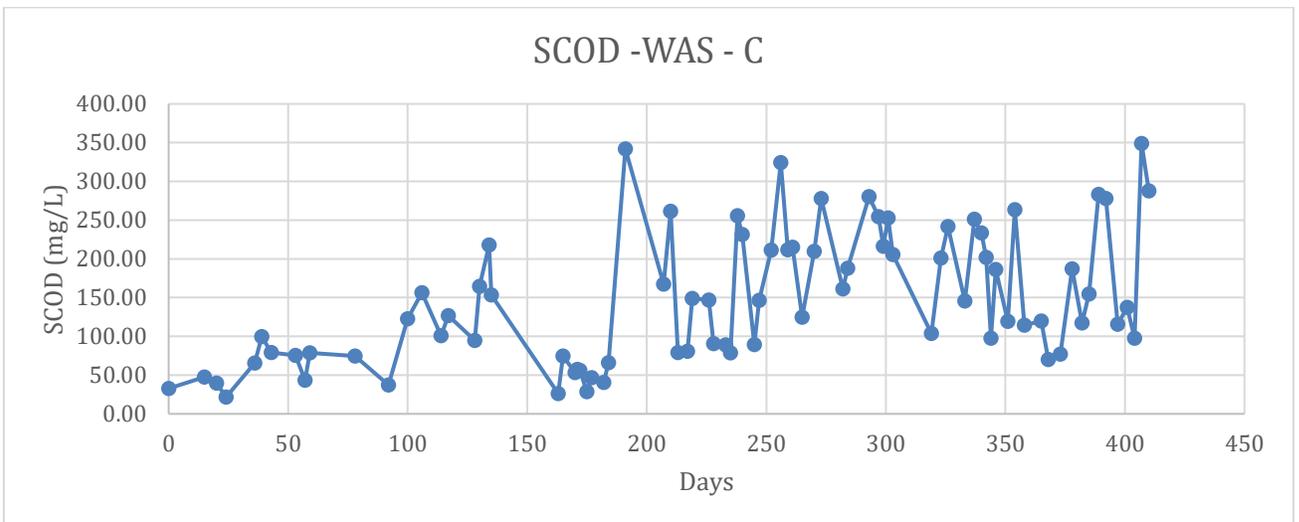
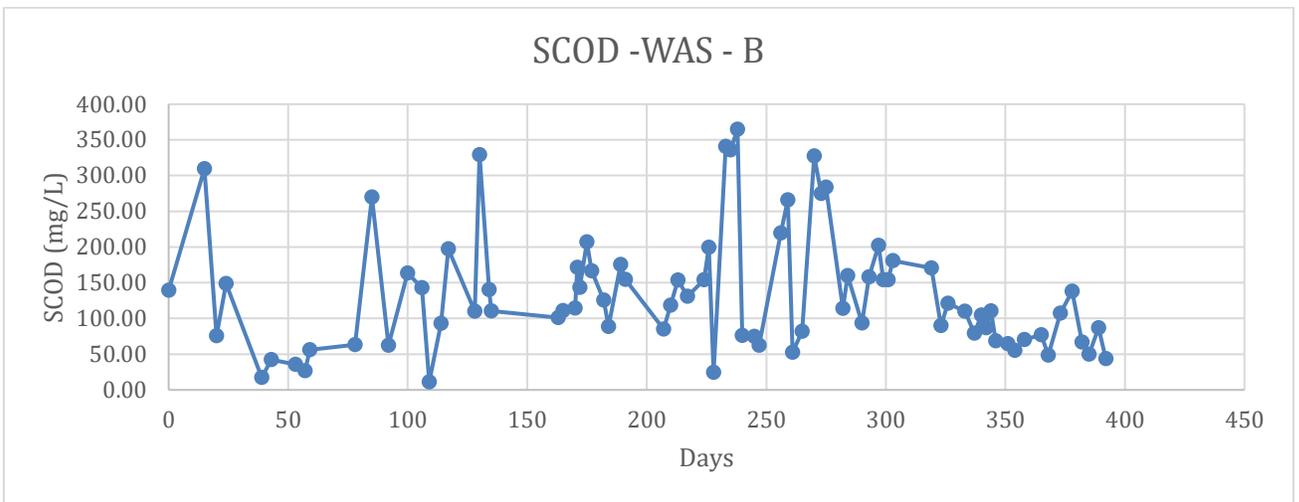
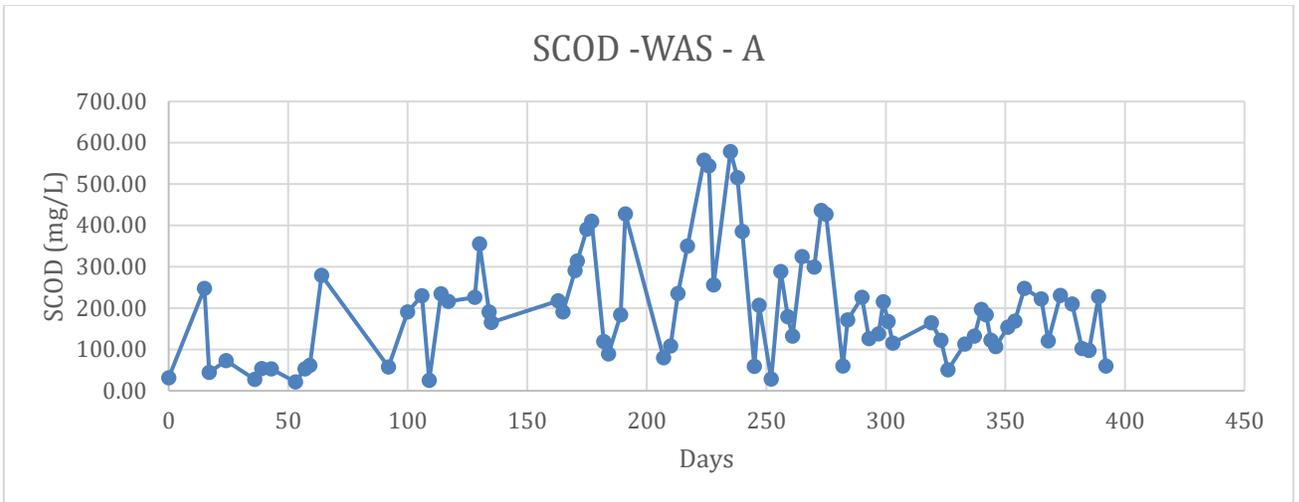
B – Raw Data





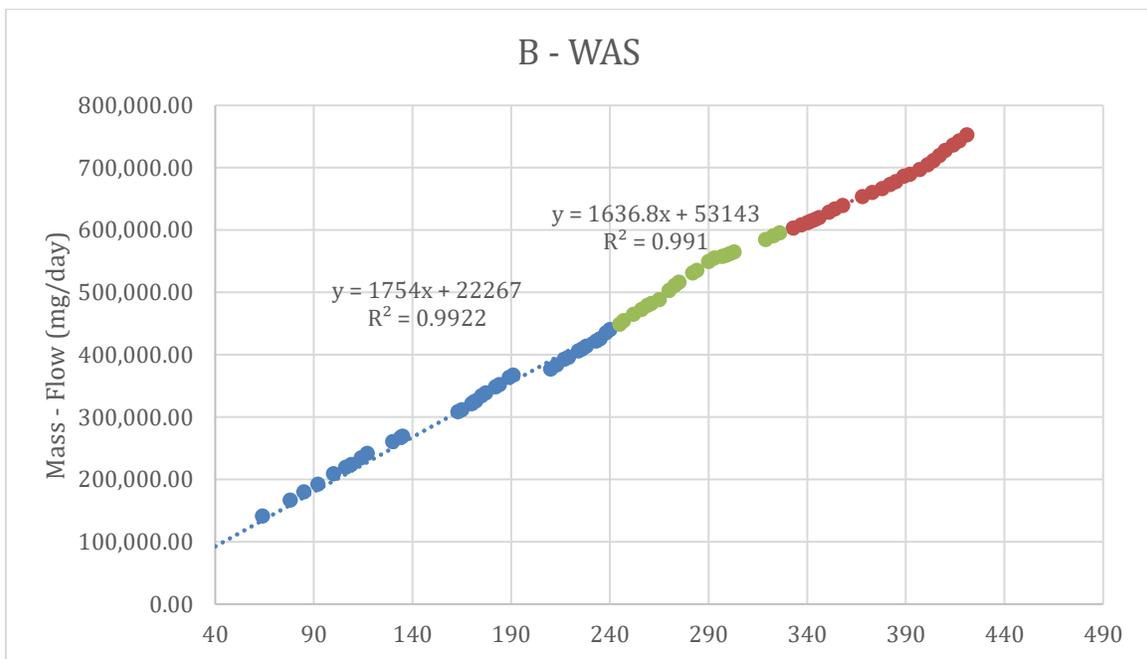
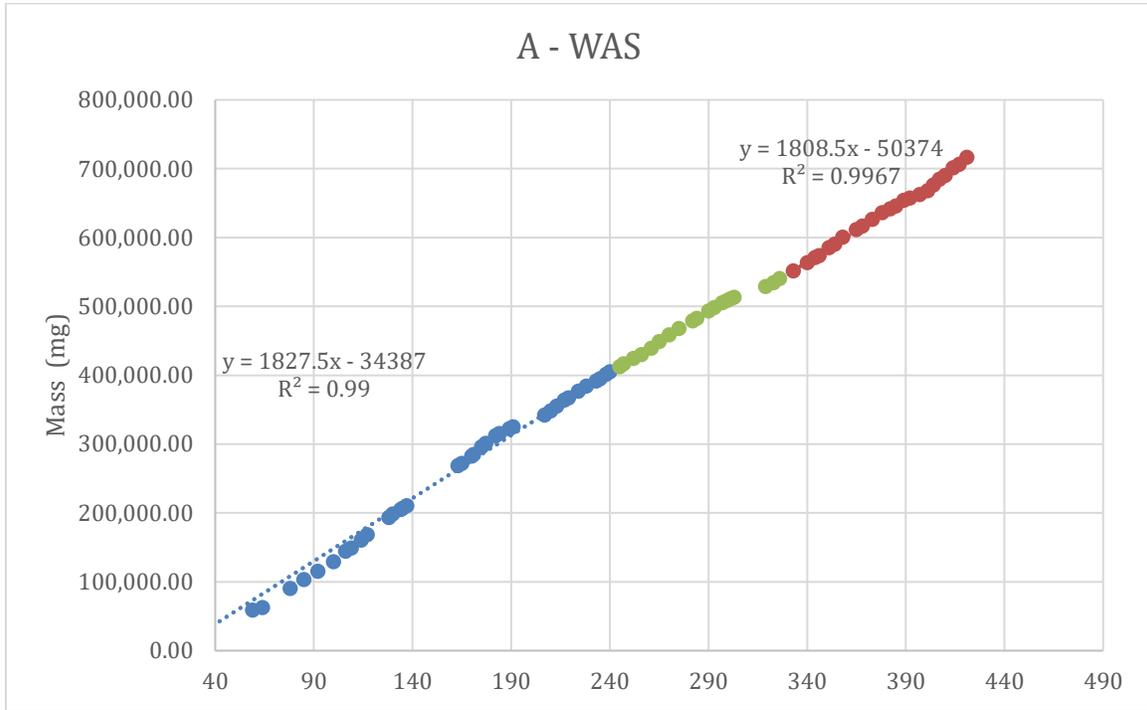


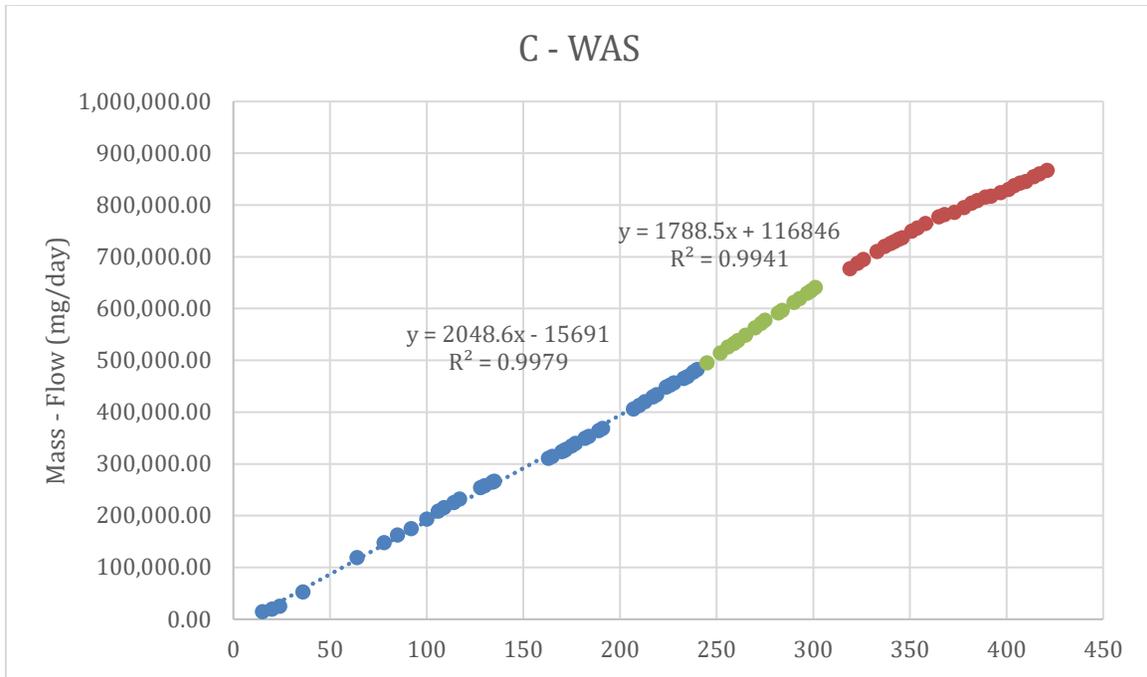




C - WAS Cumulative Data for Phase 1

C1 - Cumulative TCOD Data





Hypothesis Testing on regressions for phase 1

Regression Analysis Paratmets	A -- Regression Analysis for WAS Days 0 -241	B -- Regression Analysis for WAS Days 0 -241	C -- Regression Analysis for WAS Days 0 -241
Slope (b)	1,827.51	1,754.02	2048.584723
Standard error of the slope (sb)	29.44	25.84	15.25546464
Correlation coefficient (R^2)	0.99	0.99	0.99789713
Fishers's (F)	3,853.04	4,606.37	18032.54315
Sum of the squares of the regression	647,702,667,490.28	540,150,217,751.19	7.11398E+11
Intercept (a)	-34,386.92	22,266.60	-15690.62922
Standard error of the intercept (sa)	4,702.82	4,283.90	2535.174117
Standard error of the regression (Sy/x)	12,965.41	10,828.74	6280.984377
Degrees of Freedom (v)	39.00	36.00	38
Sum of the squares of the residuals	6,555,974,619.79	4,221,418,382.73	1499129060
Hypothesis testing			Significantly different

C2 – SCOD Phase 1 - ANOVA Single Factor

Anova: Single Factor												
SUMMARY												
Groups	Count	Sum	Average	Variance								
SCOD -WAS - A	30	8137.323	271.2441	23240.25								
SCOD -WAS - B	32	4903.074	153.2211	7365.641								
SCOD -WAS - C	30	3596.3	119.8767	6169.96								
ANOVA												
Source of Variation	SS	df	MS	F	P-value	F crit						
Between Groups	381092.6	2	190546.3	15.68455	1.46E-06	3.09887						
Within Groups	1081231	89	12148.66									
Total	1462323	91										
SD												
There IS a difference												
Tukey												
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?					
A	B	118.023	30	32	19.80658	5.95878	YES					
B	C	33.34438	32	30	19.80658	1.6835	NO					
C	A	151.3674	30	30	20.1235	7.521922	YES					
To work out critical q												
df	89											
# of group	3											
use 60	3.4											
use 120	3.36											

D – TSS VSS for Phase 1

Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
WAS -A - TSS (mg/L)	30	494925	16497.5	20458162		
WAS -B - TSS (mg/L)	30	511825	17060.83	39494340		
WAS -C - TSS (mg/L)	30	537550	17918.33	31049695		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	30714181	2	15357090	0.506265	0.604507	3.101296
Within Groups	2.64E+09	87	30334066			
Total	2.67E+09	89				
TSS no Difference						

Anova: Single Factor							
SUMMARY							
Groups	Count	Sum	Average	Variance			
WAS -A - VSS (mg/L)	25	302500	12100	12195937			
WAS -B - VSS (mg/L)	28	342800	12242.86	25561012			
WAS -C - VSS (mg/L)	26	387225	14893.27	21703928			
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	1.29E+08	2	64555603	3.216252	0.045622	3.116982	
Within Groups	1.53E+09	76	20071684				
Total	1.65E+09	78					
SD							
There IS a difference							

Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	142.8571	25	28	871.6976	0.163884	NO
B	C	2650.412	28	26	862.796	3.071887	YES
C	A	2793.269	26	25	887.3712	3.147802	YES
To work out critical q							
df	76						
# of group	3						

E – Permeate for Phase 1

Anova: Single Factor					
----------------------	--	--	--	--	--

SUMMARY							
Groups	Count	Sum	Average	Variance			
TCOD -Permeate - A	26	1346.193	51.77666	291.085			
TCOD -Permeate - B	26	1240.69	47.71884	383.8275			
TCOD -Permeate - C	29	1081.696	37.29985	327.2768			
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	3098.811	2	1549.406	4.641689	0.012455	3.113792	
Within Groups	26036.56	78	333.8021				
Total	29135.37	80					
Fcrit < F So there is a sig difference							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	4.05782	26	26	3.583091	1.132491	NO
B	C	10.41899	26	29	3.489195	2.986074	NO
C	A	14.47682	29	26	3.489195	4.149042	YES
To work out critical q							
df	78						
# of group	3						
use 60	3.4						
use 120	3.36						
Reject H0 if q > than qcrit ---- THERE IS SIG DIFFERENCE							

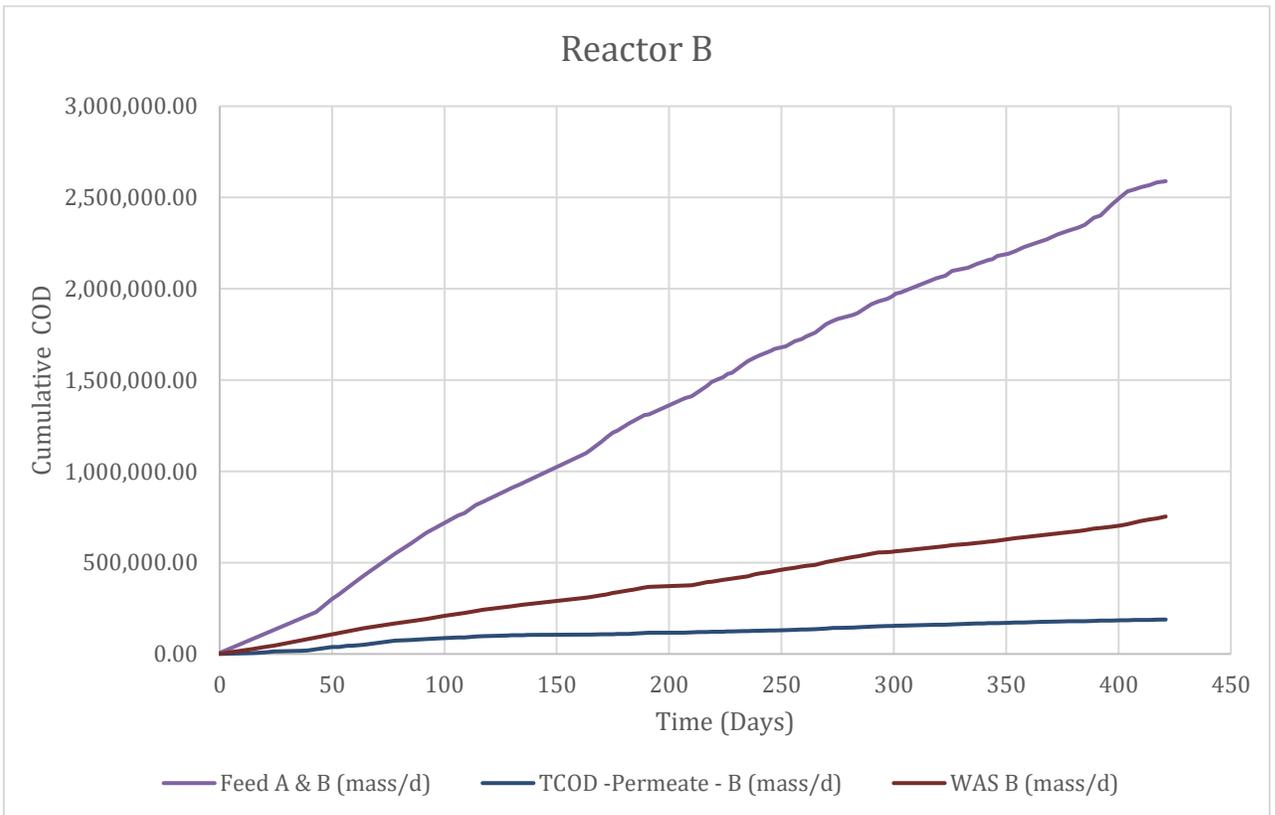
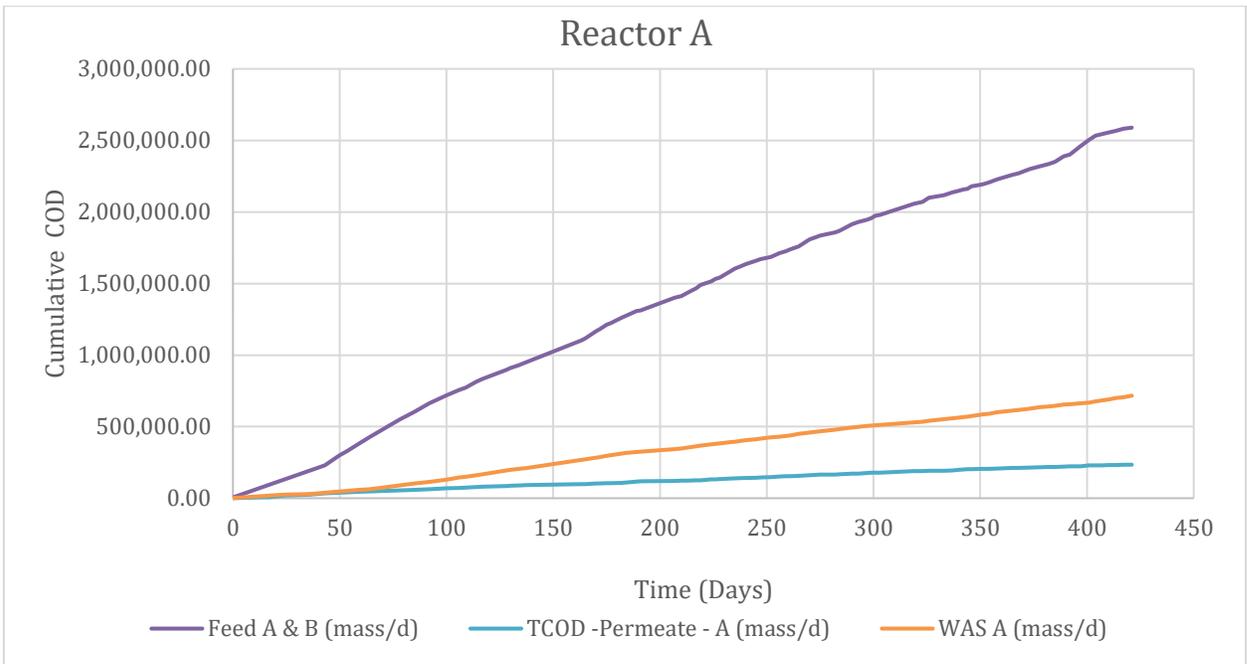
T Test: Two-Sample Assuming Equal Variances

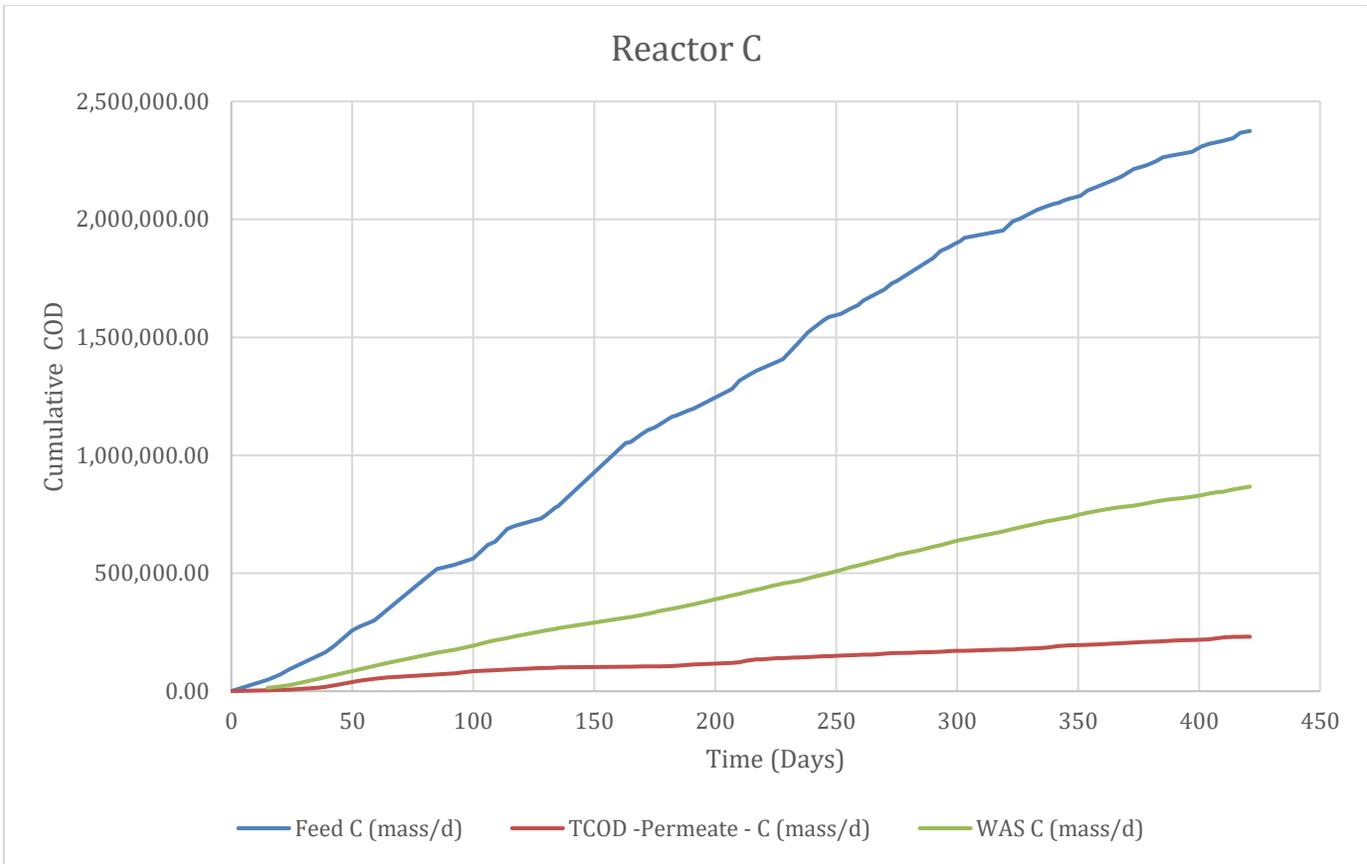
	TCOD -Permeate - A	TCOD -Permeate - C
Mean	49.74775	37.29985
Variance	335.0366	327.2768
Observations	52	29
Pooled Variance	332.2863	
Hypothesized Mean Difference	0	
df	79	
t Stat	2.946444	
P(T<=t) one-tail	0.002112	
t Critical one-tail	1.664371	
P(T<=t) two-tail	0.004223	
t Critical two-tail	1.99045	
SD		

F – Sulphate reduction calculations

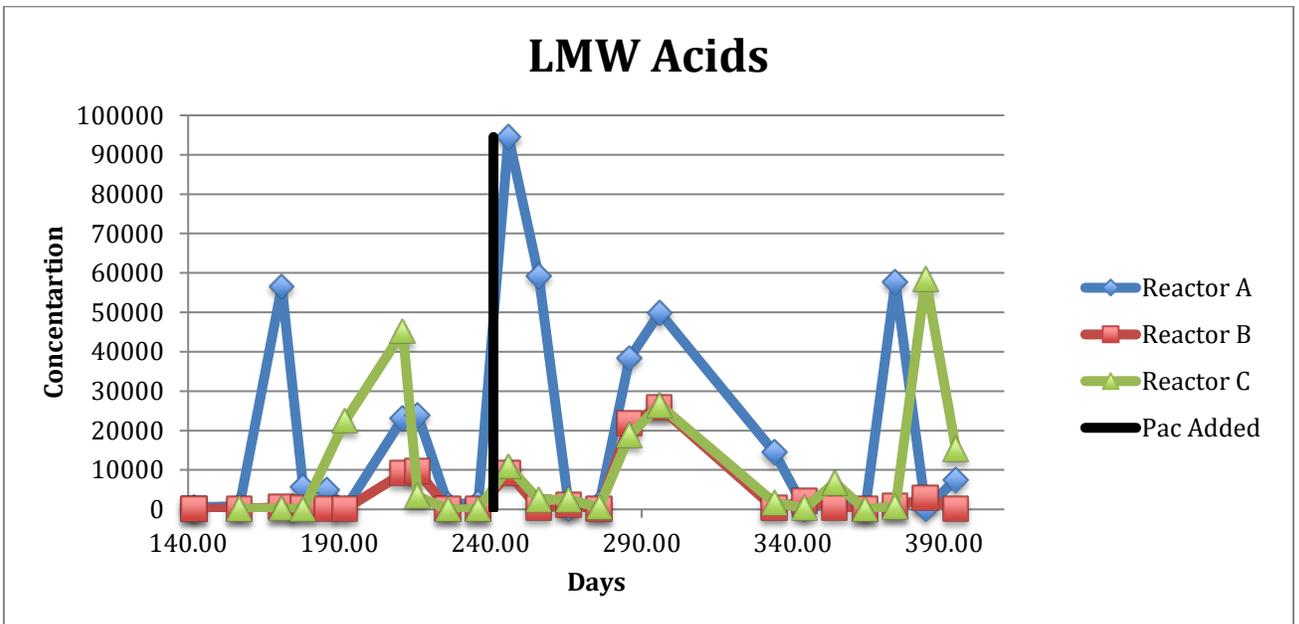
Sulfate Calculations	Value	Units
Flow	9.5	L/d
[SO ₄]	55	mg/L
COD used per SO ₄	0.67	ratio
SO ₄ per day	522.5	mg/d
COD used per Day	350.075	mg/d

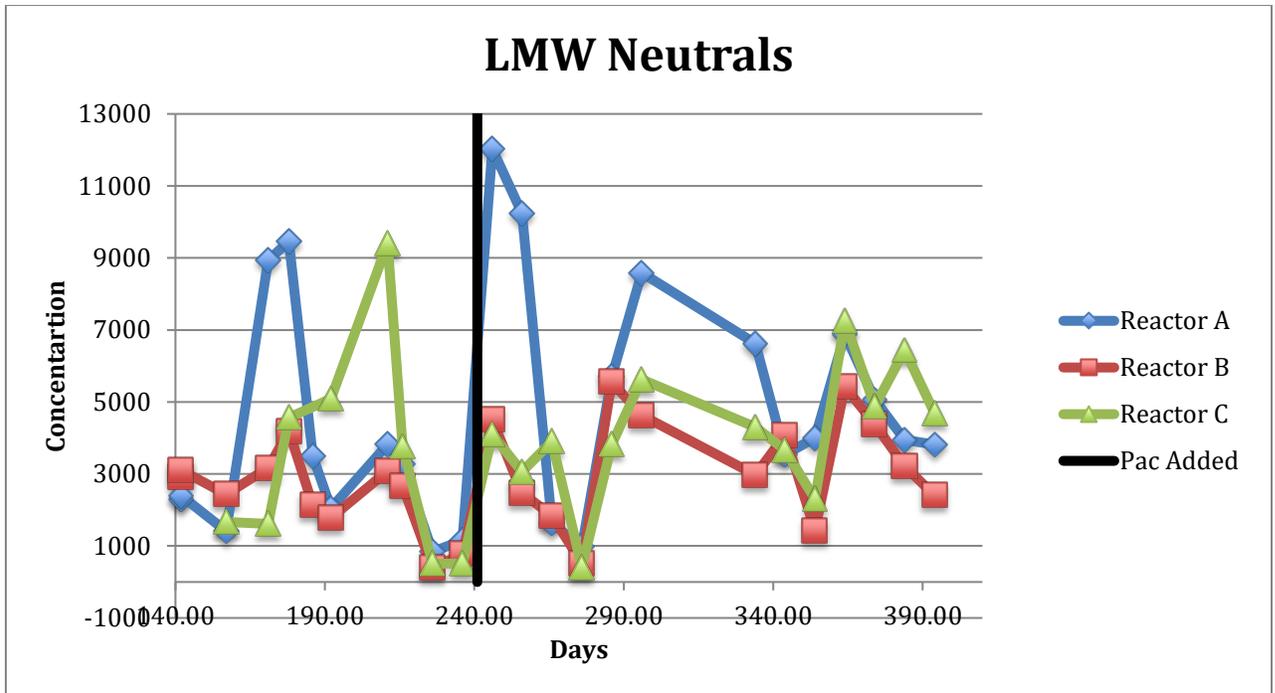
G – COD Balance for Phase 1





H – LMW form LCOCD for Phase 1





I – ANOVA for LCOCDs for Phase 1

Humics

Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	11	64500.8	5863.709	7593811		
B	10	44754.13	4475.413	1767166		
C	10	46562.68	4656.268	6312162		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	12117795	2	6058897	1.14125	0.333832	3.340386
Within Groups	1.49E+08	28	5309002			
Total	1.61E+08	30				
NO SD						

Biopolymers

Phase 1						
A - Bio-polymers	B - Bio-polymers	C- Bio-polymers				
11267.79	14792.82759	2611.862				
11009.86	15115.03448	2685.241				
8030.759	11932.68966	2727.172				
23851.72	21231.51724	8200.621				
17842.97	24113.7931	3117.793				
7585.517	7009	24650				
14638	17667	30894				
22055	14470	8709				
6870.621	1594.206897	3184.276				
9447.103	7833	13797				
Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A - Bio-polymers	10	132599.6	13259.96	37375438		
B - Bio-polymers	10	135759.3	13575.93	45933598		
C- Bio-polymers	10	100577.4	10057.74	1.03E+08		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	75772307.59	2	37886154	0.610675	0.550314	3.354131
Within Groups	1675074711	27	62039804			
Total	1750847018	29				
Fcrit > F - NO SD						

Building Blocs

Phase 1						
A - Building B	B - Building B	C- Building B				
1085.586	2962.758621	902.6207				
1406.483	2979.310345	793.931				
8135.448	729.3793103	989.2414				
7906.897	2151.586207	534.4138				
4185.31	2252.482759	1524.414				
987.931	918	3526				
4003	2767	10023				
3543	3131	1618				
695.7241	305.3793103	350.3448				
2575	1762	1751				
Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A - Building B	10	34523.9	3452.39	7427998		
B - Building B	10	19958.79	1995.879	1061346		
C- Building B	10	22012.93	2201.293	8364127		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	12429532.64	2	6214766	1.106259	0.345326	3.354131
Within Groups	151681235.9	27	5617824			
Total	164110768.5	29				
Ferit > F - NO SD						

J – Validations of Phase 1 and Phase 2 data

It was deemed to be important to validate the lab data to identify if there are any systematic errors, random errors or blunders. The data set can be subjected to two main checks

Check 1

It was expected that the concentration of $SCOD_{WAS}$ should be greater than the concentration of the $TCOD_{Per}$. This check was used to validate the data set and identify any errors in the data for each reactor.

Check 2

It was expected that the TCOD in WAS is proportion to VSS since it mostly consists of suspended solids. A typical ratio for bacterial biomass is 1.42 gCOD/g VSS, in which case the biomass is assumed to be fully-grown. Thus a TCOD can be calculated for the WAS and it can be compared to the measured TCOD in the WAS. However, the organic content in the feeds varies and based on the composition the oxidation rate will range, thus the gCOD/g VSS will also change. This can fluctuate from 1.3 – 1.9, so while comparing this should be considered.

$$TCOD = pCOD + SCOD \text{ (Eq 1: Gori , 2014)}$$

$$pCOD = 1.42VSS \text{ (Eq 2: Gori , 2014)}$$

$$TCOD_{cal} = 1.42*VSS + SCOD \text{ (Eq 3)}$$

Reactor A

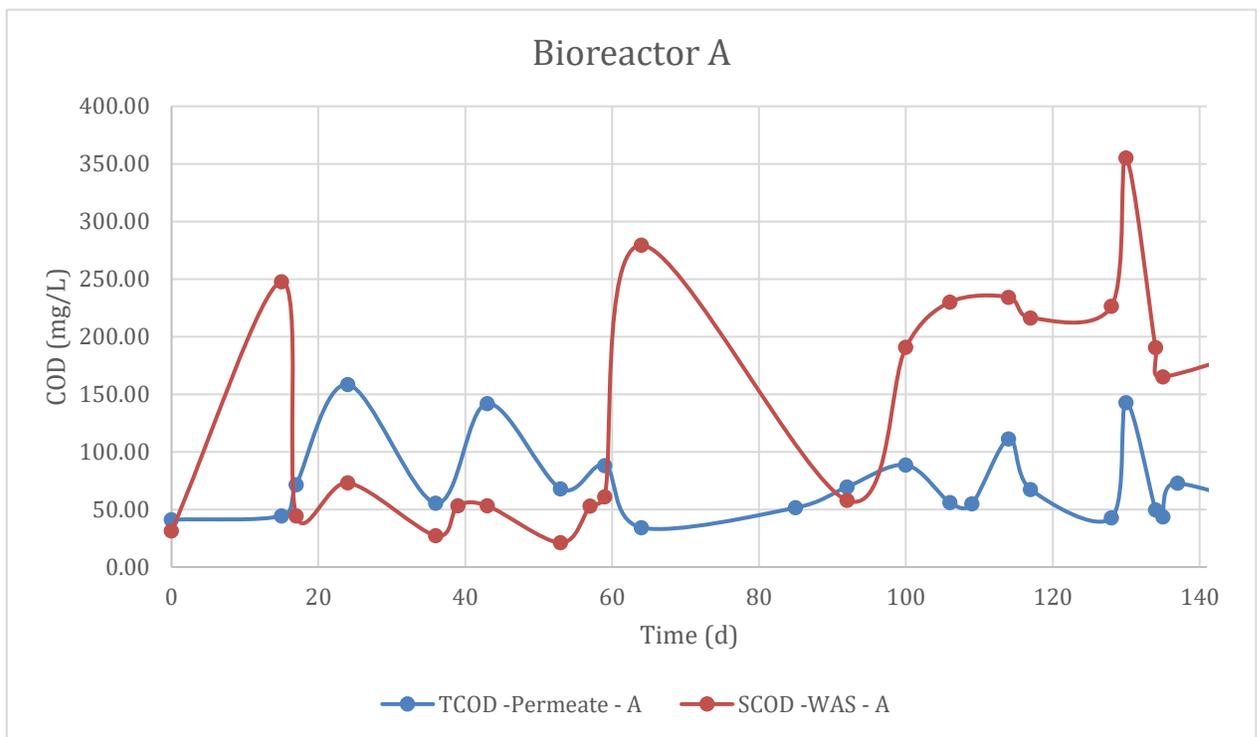
Check 1

Figure 4.7 illustrates the $SCOD_{WAS}$ and $TCOD_{Per}$ for reactor A. Based on the initial check ($SCOD_{WAS} > TCOD_{Per}$) it is clear to see that the SCOD line is always above the TCOD line. From the 17th day to the 59th day the SCOD line has dipped below the TCOD which does not make sense as the membrane pores are smaller than the filter paper used to conduct the SCOD test, as explained earlier.

There are two possible explanations to this. There could be a tare or hole in the membrane that suctioned the larger organics through the membrane or the SCOD test was executed incorrectly. One can observe the blue line closely and see that it is consistent even during the fourth two days with just two peaks, which are still under 200mg/L making it

within the general range of the rest of the data. This leads to the conclusion that the second reason is more likely and that the SCOD tests were conducted inaccurately.

When observing the SCOD line from day 59 onwards, it is clear to see that the peaks that occur, occur gradually. As in, once there is a sudden decline the increase is gradual until the next peak. This validates the points as the increase and decrease is visible through the days. It is not as if there is a random hike in the reactor. This was observed though out the testing period expect for few exceptions. In addition it was determined that steady state was reached after day 90. So the invalidity of the data points before day 90 does not

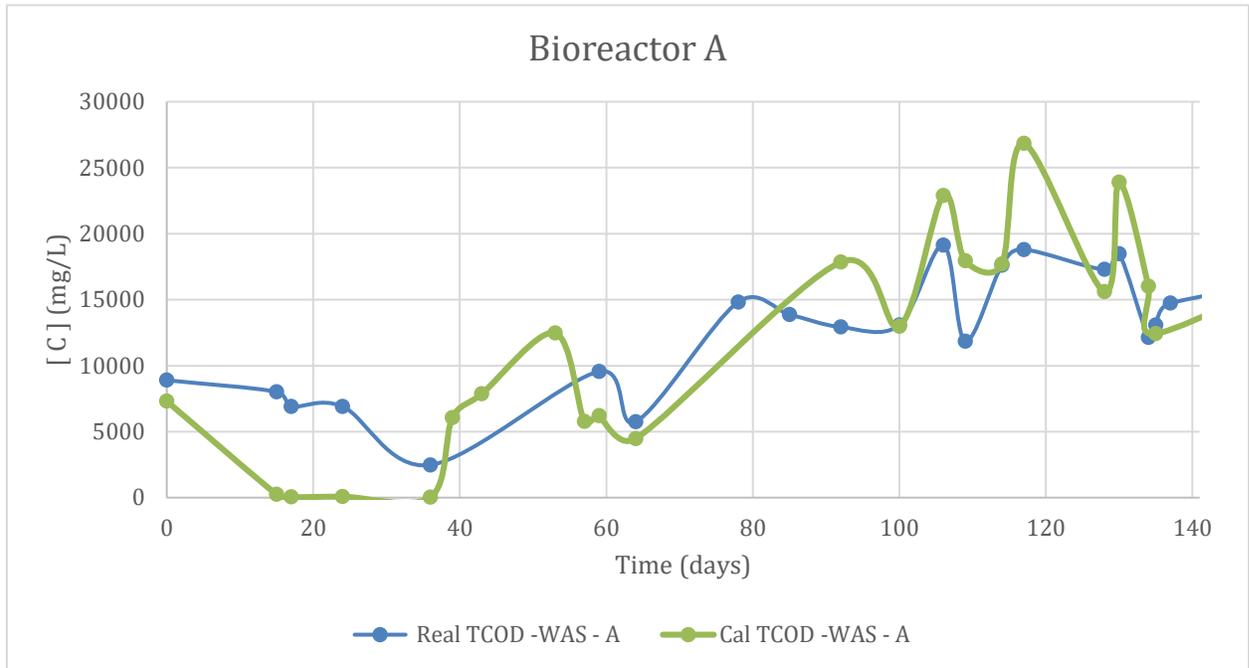


influence the analysis of phase 1.

Figure 4. 26: Check 1 for reactor A in phase 1

Reactor A Check 2

In order to subject the data collected from reactor A to the second check, a TCOD can be calculated based on the VSS and SCOD by rearranging the equations to give the equation for calculating TCOD. This has been mapped out as shown in Figure 4. 27. From looking at Figure 4. 27 it is very evident to see that both the lines follow almost identical patterns. The pattern displayed in the calculated TCOD and real TCOD are identical, as in both fluctuate in the same manner. In addition both are similar in terms of value. This



validates the data for reactor A in phase 1.

Figure 4. 27: Check 2 for reactor A in phase 1

Reactor B

Reactor B – Check 1

As both the checks were subjected towards reactor A, similarly it can be applied to B. Figure 4.9 illustrates the $TCOD_{per}$ and $SCOD_{WAS}$ to determine if any TCOD readings from the permeate sample is higher. ($SCOD > TCOD_p$)

Looking at the Figure 4. 28 it is clear to see that the SCOD is always greater than the TCOD except for the day 36th to 78th. This is the similar pattern observed in reactor A too. As explained earlier this is not theoretically correct as the membrane pores are smaller. So the same two explanations are viable, as suggested for A. Looking slightly ahead, the same pattern can be seen in reactor C around the same time. It is highly unlikely that that all 3

reactors had a hole or tare that repaired itself. The fact that this error appears in all three reactors ensures that it is highly likely that the SCOD test were executed inaccurately and for this reason these data points were excluded. Moreover as explained earlier, steady state was only established at day 90 so these data points are insignificant in as they are not included in the analysis of phase 1.

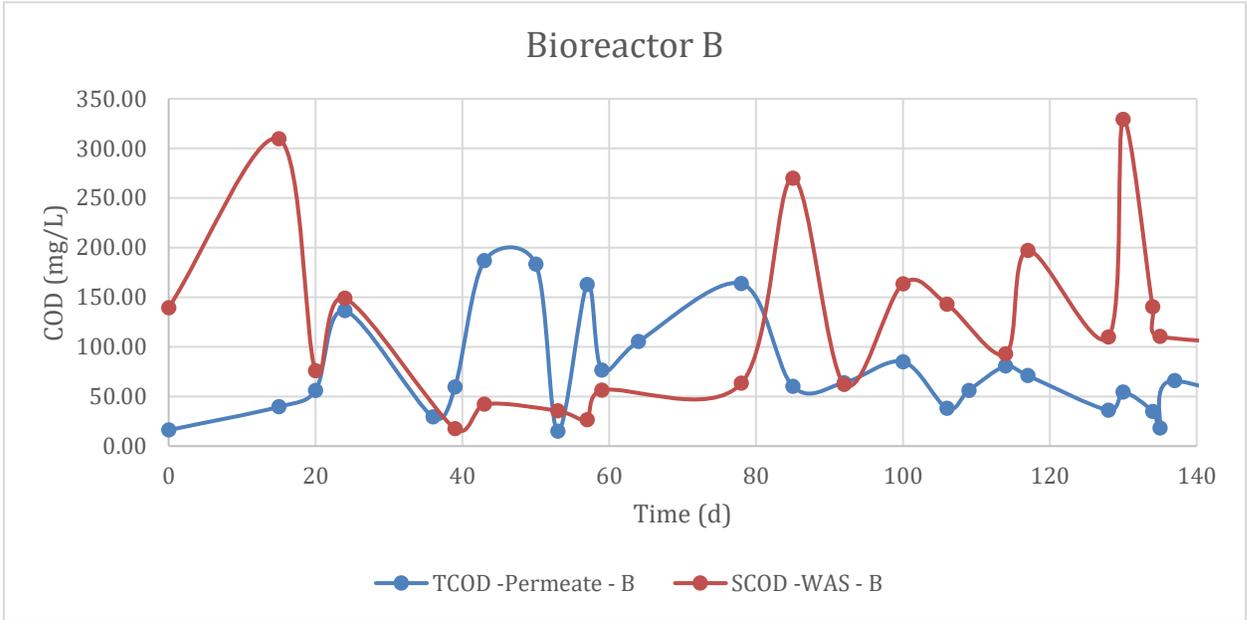


Figure 4. 28: Check 1 for reactor B in phase 1

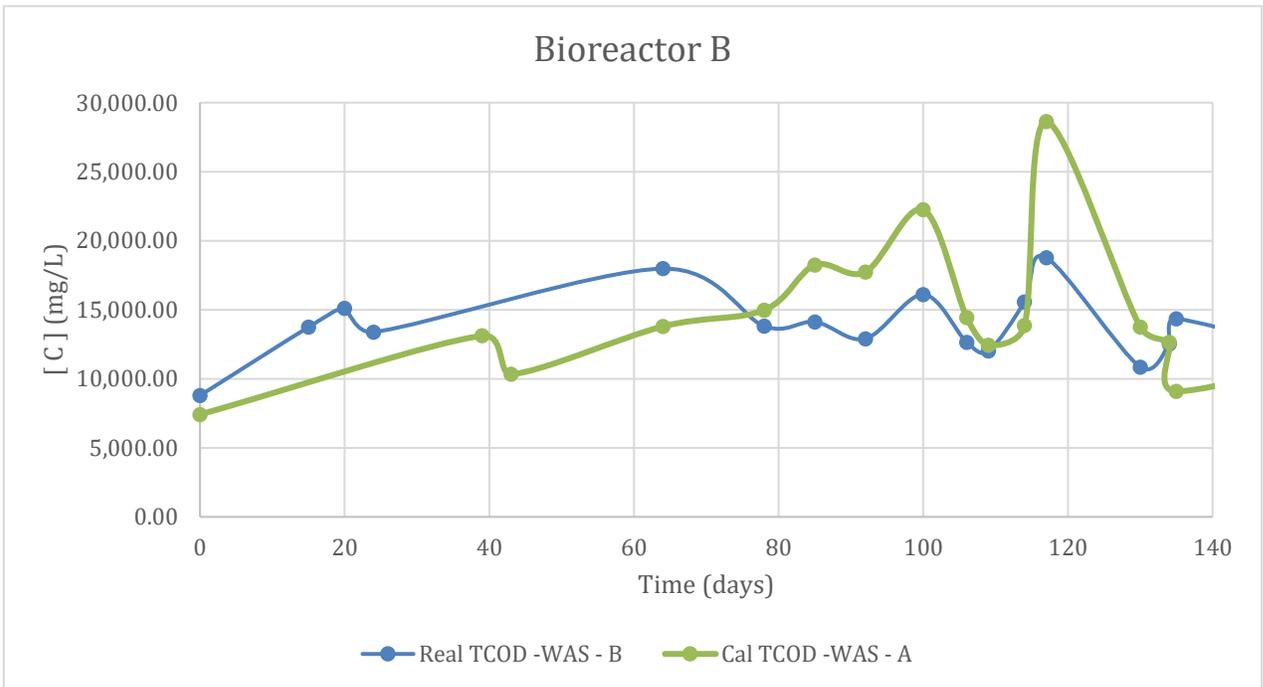


Figure 4. 29: Check 2 for reactor B in phase 1

Reactor B – Check 2

The second check can also be applied to the reactor as show in

Figure 4. 29. From this it is evident that both the real and calculated TCOD is fairly similar overall. In addition after day 64 the fluctuations of both calculated and real TCOD are almost identical, the ratios are slightly different. For instance, on day 85 both the calculated and actual COD rises and on day 92 they both drop. However, actual TCOD decreases by 9% where as calculated TCOD decreases by 3%. This is anticipated as the organic compositions is not consistent. The presences of increase fats and proteins can alter the constant 1.42, making the calculated TCOD an inaccurate representation of the measured TCOD. Over the duration of phase 1 the fluctuations are synchronized and the difference between the two is less than a magnitude of 2 so it can be concluded that the data in phase 1 is validated.

Reactor C

Reactor C – Check 1

Figure 4. 30 illustrates the TCOD concentration in the permeate and the SCOD concentration in the WAS and very similar to the last two reactors. The pattern is identical. After the initial 100 days the fluctuations in the permeate COD decreases significantly as most of the readings are below 50mg/L. The TCOD levels in the WAS continue to fluctuate between 120mg/L to 220mg/L. Overall the fluctuations are similar with the permeate values being less drastic. In addition, since day 92, the $SCOD_{WAS}$ is always higher than the $TCOD_{per}$ therefore; the data set can be deemed as a valid set.

Figure 4. 31 displays the real TCOD and the calculated TCOD (calculated as explained in section 4.2.2). It is clear that both the values are very similar as they are often superimposed on to each other for the first 191 days. After which the magnitude is off by 1.4. This is still acceptable as this could be due to changes in the feed composition. Especially, during days 90 – 191 the fluctuation between both TCODs are identical and the values are very similar. This can verify the methods used to sample and collect data for reactor B is valid.

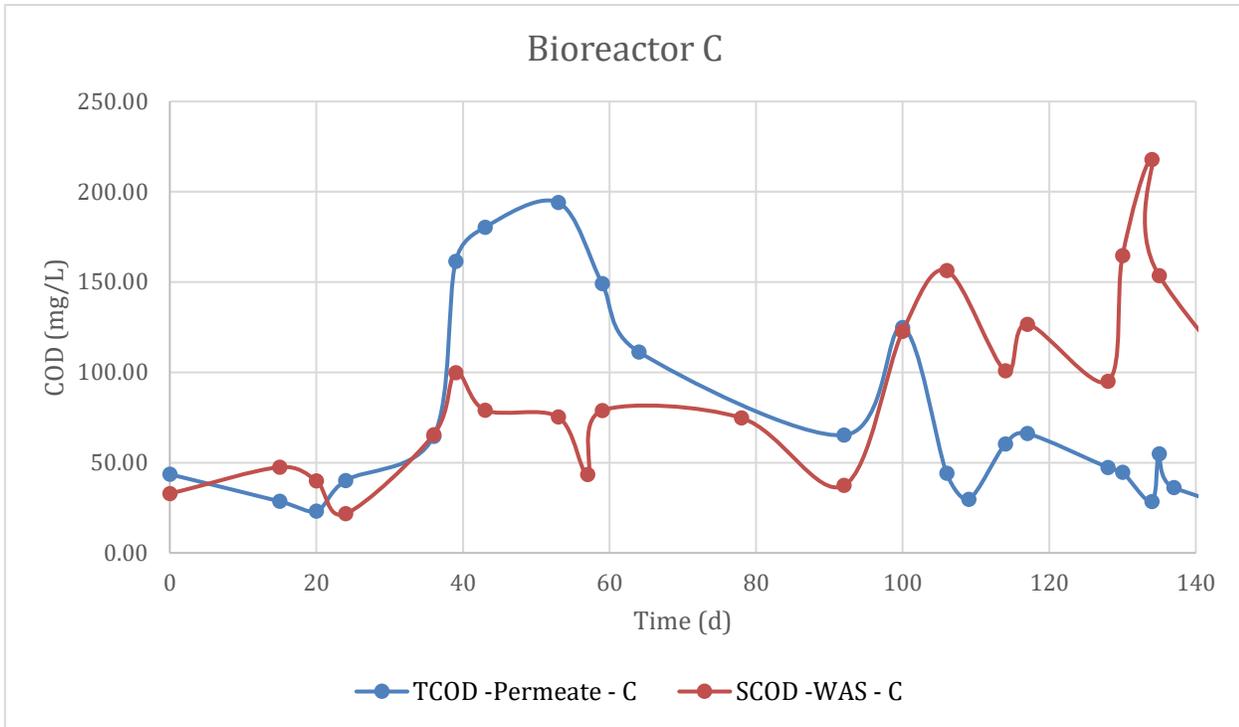


Figure 4. 30: Check 1 for reactor C in phase 1

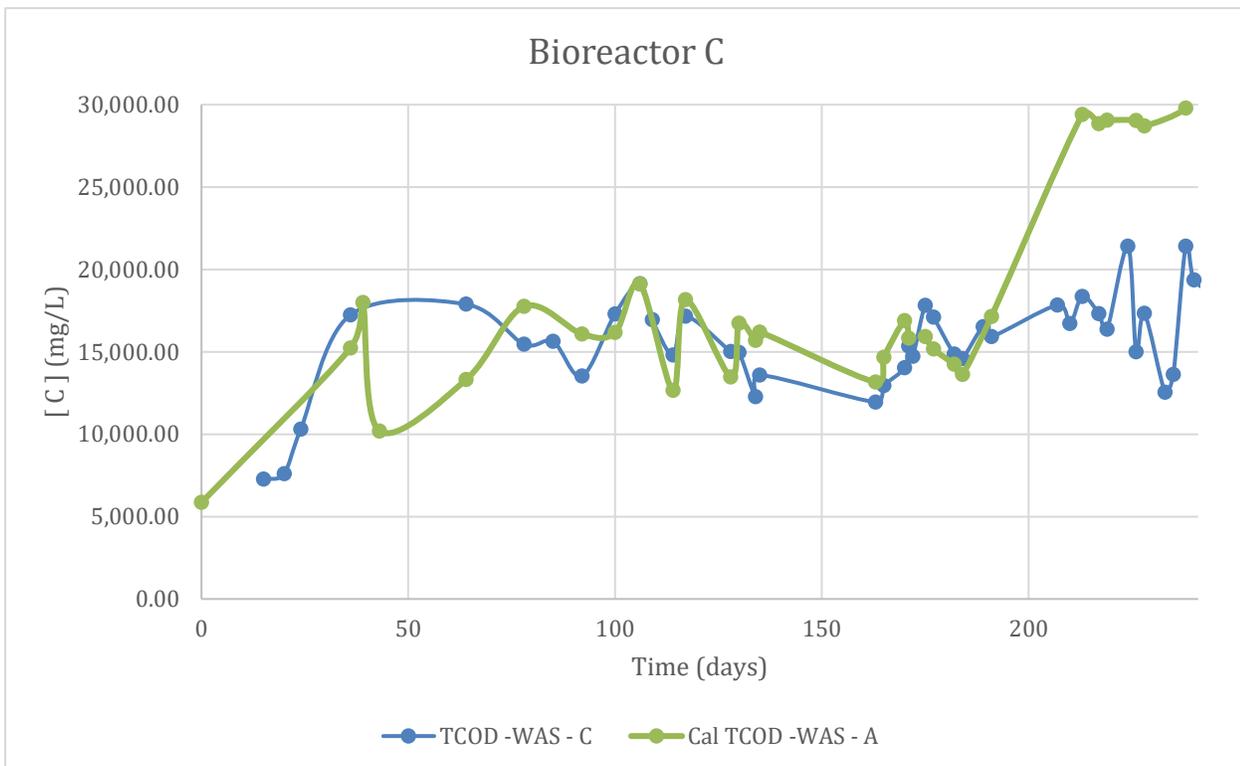


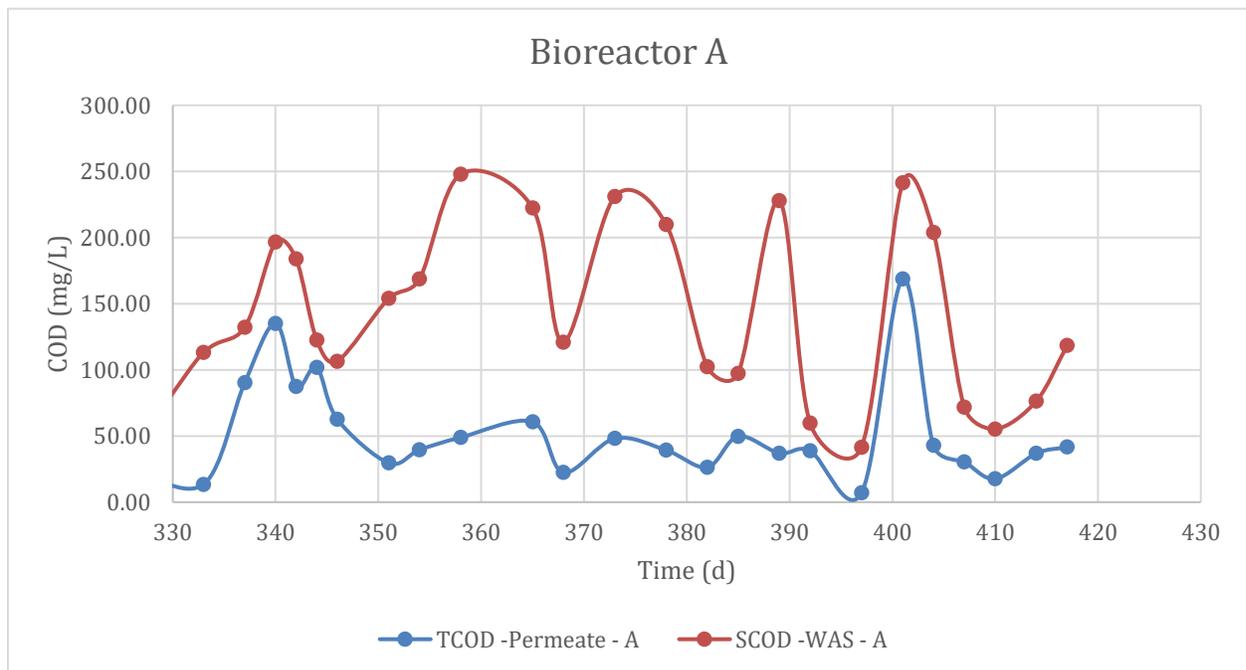
Figure 4. 31: Check 2 for reactor C in phase 1

Validate Phase 2 Data

As explained in section 4.2.4 it is very important to validate data when feasible and this can be done using the same checks applied in the phase 1 of the experiment. The concentration of SCOD_{WAS} should be greater than the concentration of the TCOD_{Per} based on the pore size of the membrane (0.04 μm) and the filter paper used to measure the SCOD_{WAS} (1.5 μm). This can be applied to each reactor separately. In addition the description provided in section 4.2.2 indicates that the TCOD can be calculated based on SCOD and VSS, both the calculated and experimental CODs be compared to see if there are specific values that do not fit the pattern and range. In this section, each reactor's data was validated based on these checks.

Reactor A

The validation check can be applied to reactor A by superimposing SCOD_{WAS} and TCOD_{Per} results on a graph as shown in Figure 4. 32 From it can be seen that the COD in the permeate is always below the SCOD in the WAS. In addition it is interesting to note they both follow the same fluctuations. For instance on day 397 both the SCOD_{WAS} and TCOD_{Per} experience a drop to 41.59 and 7.09 respectively and it is followed by a hike on day 401 to 241.36 and 168.76 as the system recovers for this drop. This is an increase of approximately 200mg/L in both. This type of pattern can be seen throughout the testing phase, hence it is



safe to conclude that the data for reactor A is valid.

Figure 4. 32: Check 1 for Reactor A in phase 2

In order to apply the second check to test reactor A both the calculated and the actual $TCOD_{WAS}$ was plotted on a graph to see how the data correlated to each other as can be seen on the Figure 4. 33. From this it is interesting to note that they both follow the same pattern, as in the dips and the hikes are almost identical in both sets. However, they are off by a magnitude of 1.43 on average. This can be explained due to the composition of the of the feed. The TCOD was calculated on based on the assumed that the feed contains fully grown biomass, whereas the feed could have a non typical ratios of lipids to protein to carbohydrates ratio. This variation will alter the organic oxidation rate thus this brings about a magnitudinal difference in the calculated and actual TCOD data

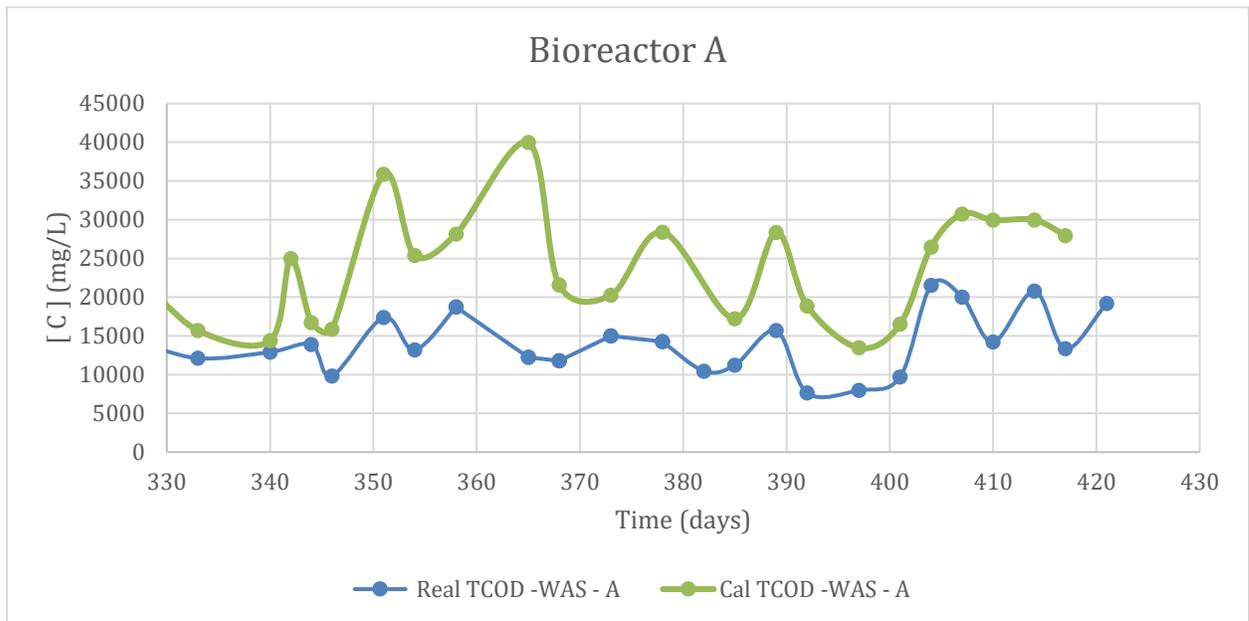


Figure 4. 33: Check 2 for reactor A in phase 2

Reactor B

Initially by observing Figure 4.27 it is very evident that the $SCOD_{WAS}$ is always greater than $TCOD_{per}$. In addition majority of the time the fluctuations are also synchronized with each other except for the three points that have been circled in red. The values for the SCOD seem to be a little high in comparison however, not enough to disregard the points as they are still within the expected range of SCOD.

To implement the second check on data sample for reactor B the same technique can be used. So the both the calculated and actual TCOD_{WAS} are plotted on

Figure 4. 35. Similar to reactor A the fluctuation are the same in both TCOD values. However, similar to reactor A in phase 2 both TCOD are off by a magnitude. As defined earlier this further enforces that this could be due to the change in feed composition mentioned earlier as both reactor A and B display the same pattern and it can be expected to be evident in reactor C as can be seen later on in this section.

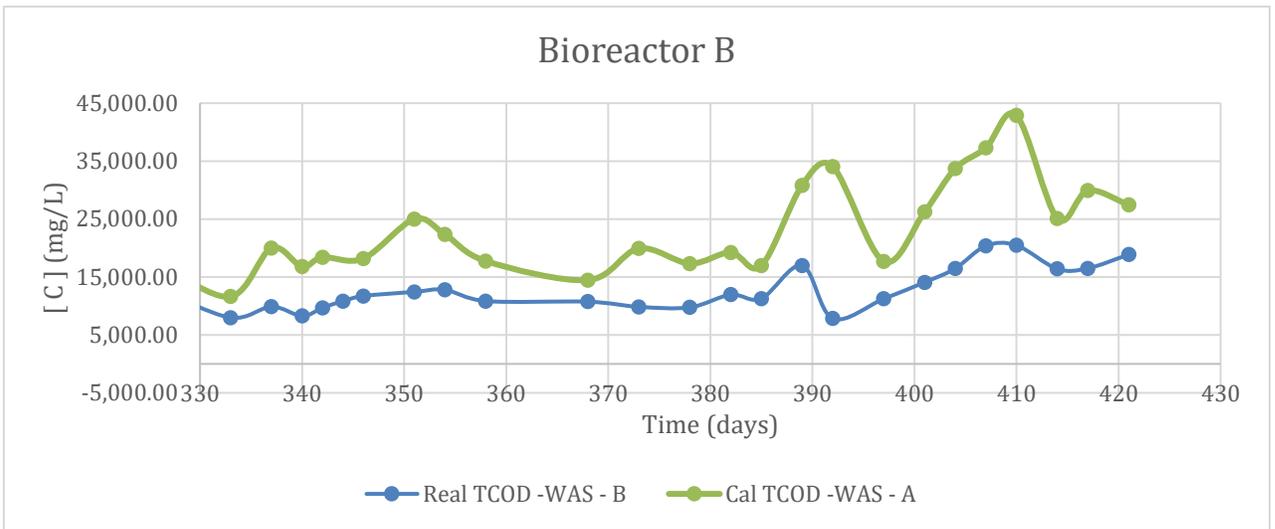


Figure 4. 34: Check 1 for reactor B in phase 2

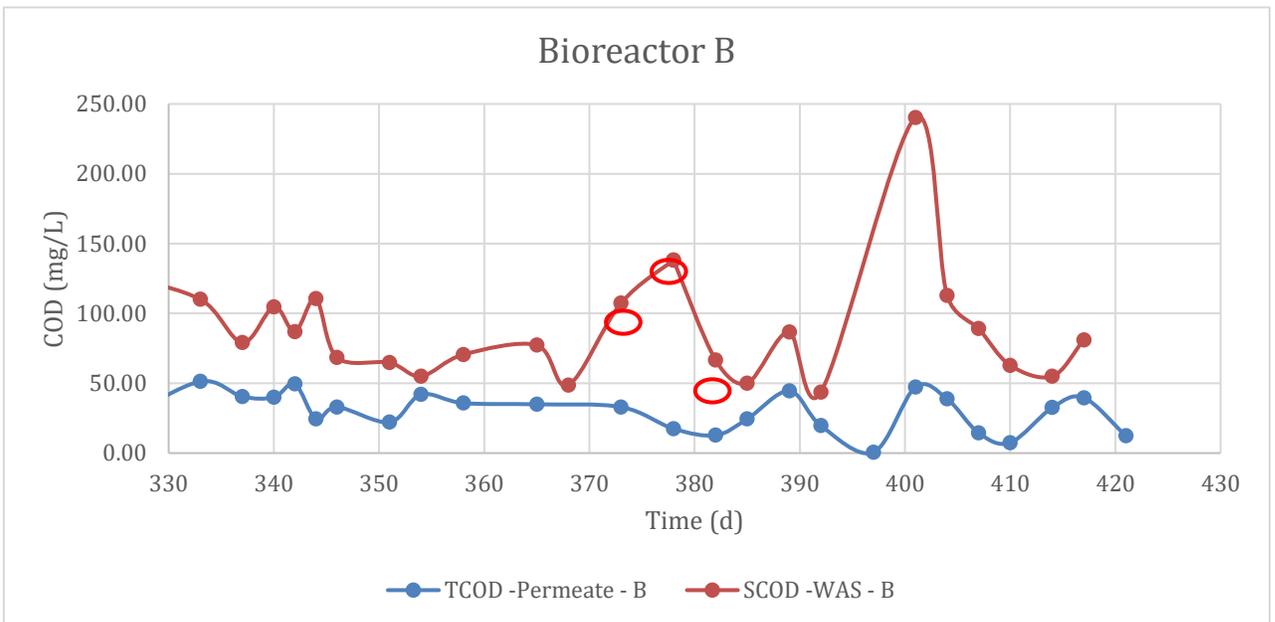
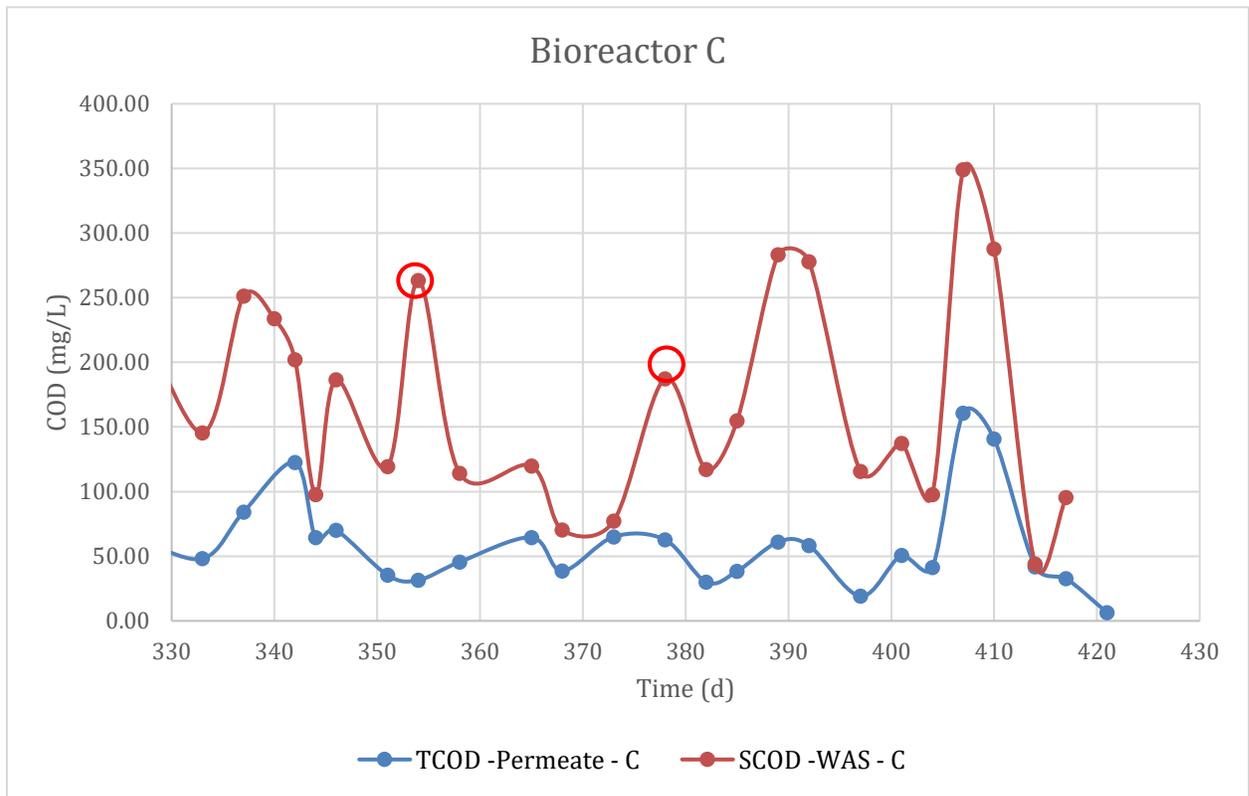


Figure 4. 35: Check 2 for reactor B in phase 2

Reactor C

Figure 4.11 shows the SCOD in the WAS and the TCOD in the permeate are plotted, in order to apply the first check. The SCOD in reactor C is approximately five times (4.8) higher than the TCOD in the permeate during phase 2 of experimentation. This ensures that there is no tare or rip in the membrane. In addition it is suggestive that there are no major errors in the data based on the pore sizes. Similar to the other two reactors the fluctuations observed in the permeate are directly correlated to the WAS concentration with a few exceptions that have been highlighted with a red circle in Figure 30. This is anticipated as the permeate is filtered WAS. In addition the magnitude in WAS is higher partly due to the



complex biopolymers in the WW and partly due to the particulates of PAC.

Figure 4. 36: Check 1 for reactor C in phase 2

The second check can also be applied to reactor C to ensure that the data collected is an accurate representation of the conditions inside the reactor and the quality of treatment. As can be seen from Figure 4.31 the actual TCOD inside the reactor follows the identical pattern to the calculated TCOD at a lower magnitude of 1.78. All the fluctuation as are mirrored in each other just like the other two reactors. The exceptions have been indicated

with a red circle. This is off by a magnitude of 1.78, which further enhances the theory said earlier as it is a pattern that is evident in all three reactor during phase two.

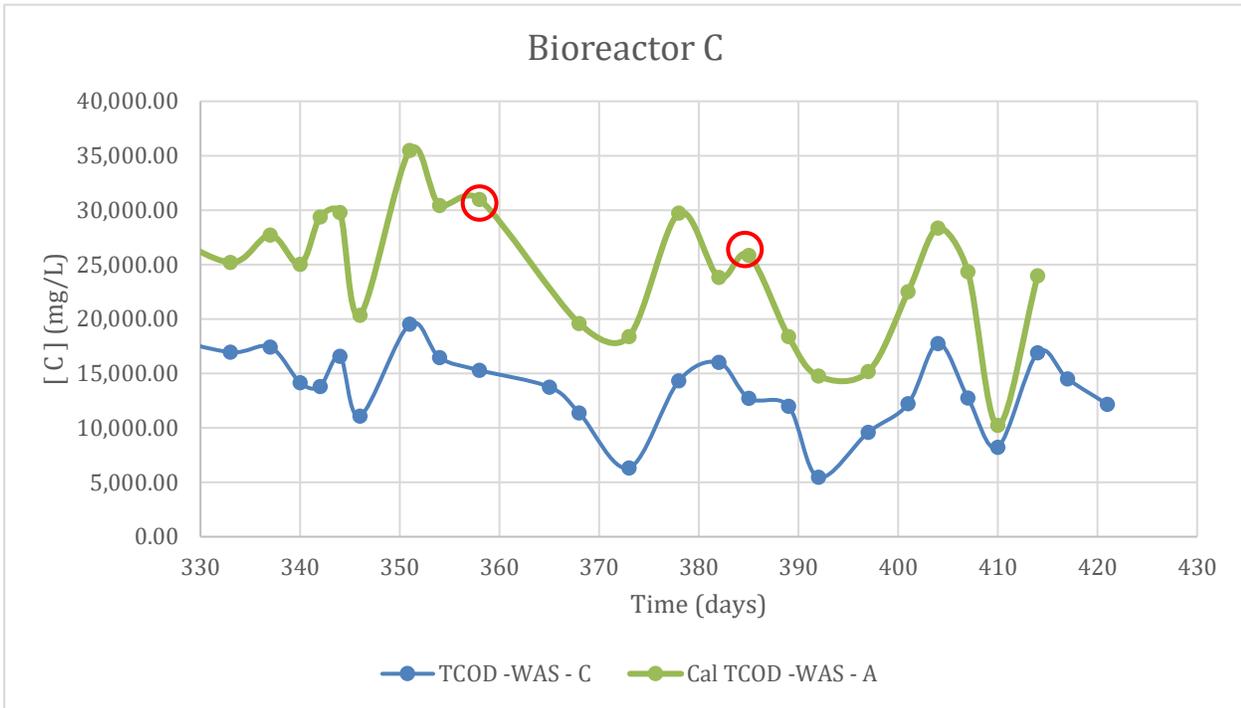


Figure 4. 37: Check 2 for reactor C in phase 2

Conclusion

All three reactors have a SCOD that always greater than the COD in the permeate on the specific given day. Also, the $TCOD_{cal}$ is greater than the actual TCOD, which can be explained by the feed composition as they are both off by a magnitude of 1.7-1.75mg/L. Additionally, when analyzing the feed in section 4.1 it was concluded based on the T test that there was a significant difference between feed in phase 1 compared to phase 2. This could be due to seasonal variations which intern could alter the carbohydrate to protein to lipids ration in the feed, this in turns modifies the oxidation rates. The VSS/COD constant in Equation --- will no longer be 1.42 thus the calculated TCOD is off in all three reactors. So over all it can be conclude that the data collected in phase 2 is an accurate representation of the condition inside the bioreactor and the quality of treatment.

K – TMP

Raw Data

Sample of raw data

Days	A (TMP - Hg)	B (TMP - Hg)	C (TMP - Hg)
262.0072569	5.1627	4.7699	4.6153
262.0079514	5.1627	4.7699	4.5862
262.0086458	5.1627	4.7247	4.5724
262.0093403	5.1627	4.7198	4.5711
262.0100347	5.1627	4.8273	4.5761
262.0107292	5.1627	4.9176	4.5862
262.0114236	5.1627	4.8676	4.6405
262.0121181	5.1627	4.7369	4.6481
262.0128125	5.1627	4.7467	4.6001
262.0135069	5.1627	4.8053	4.59
262.0142014	5.1627	4.7955	4.8007
262.0148958	5.1627	4.7577	4.8032
262.0155903	5.1627	4.7699	4.8032
262.0162847	5.1627	4.754	4.8045
262.0169792	5.1627	4.7992	4.8057
262.0176736	5.1627	4.9152	4.8057
262.0183681	5.1627	4.8859	4.8045
262.0190625	5.1627	4.8029	4.6127
262.0197569	5.1627	4.8114	4.6165
262.0204514	5.1627	4.7943	4.5951
262.0211458	5.1627	4.7711	4.6064
262.0218403	5.1627	4.7125	4.5875
262.0225347	5.1627	4.7687	4.5761
262.0232292	5.1627	4.776	4.5661

262.0239236	5.1627	4.8163	4.6102
262.0246181	5.1627	4.9127	4.619
262.0253125	5.1627	4.8712	4.6304
262.0260069	5.1627	4.7614	4.7868
262.0267014	5.1627	4.7711	4.7956
262.0273958	5.1627	4.8224	4.7994

MatLab Code

```

a_data_copy = cdata;
outlier_index = isoutlier(a_data_copy(:,2));
a_data_copy(outlier_index,2) = mean(a_data_copy(~outlier_index,2));
[upperpk,upperloc] = findpeaks(a_data_copy(:,2), 'MinPeakProminence',0.08);
[lowerpk,lowerloc] = findpeaks(-a_data_copy(:,2), 'MinPeakProminence',0.08);
moving_mean = movmean(a_data_copy(:,2), 50);
plot(a_data_copy(:,1), a_data_copy(:,2))
hold on
%plot(a_data_copy(upperloc,1), a_data_copy(upperloc,2), 'r')
%plot(a_data_copy(lowerloc,1), a_data_copy(lowerloc,2), 'k')
%plot(a_data_copy(:,1), moving_mean, '-y')
xlabel('Time(Days)')
ylabel('TMP(Arbitrary)')
title("C - Raw")

figure
plot(a_data_copy(upperloc,1), a_data_copy(upperloc,2), 'r')
xlabel('Time(Days)')
ylabel('TMP(Arbitrary)')
title("C - Highs")

figure
plot(a_data_copy(lowerloc,1), a_data_copy(lowerloc,2), 'k')
xlabel('Time(Days)')
ylabel('TMP(Arbitrary)')
title("C - Lows")

figure
plot(a_data_copy(:,1), moving_mean, '-b')
xlabel('Time(Days)')
ylabel('TMP(Arbitrary)')
title("C - Moving Mean")

figure
[idX, C] = kmeans(a_data_copy,25);
plot(a_data_copy(:,1), a_data_copy(:,2))

```

hold on
 plot(C(:,1),C(:,2),'kx',...
 'MarkerSize',15,'LineWidth',3)

L - WAS Calculations for Phase 2

Anova: Single Factor - TCOD						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	29	408083.5782	14071.84752	13689258		
B	24	297665.8628	12402.74428	14874133		
C	25	337320.163	13492.80652	12483084		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	37115588.21	2	18557794.11	1.35789	0.263452	3.118642
Within Groups	1024998291	75	13666643.88			
Total	1062113879	77				
NO SD						

Anova: Single Factor -SCOD						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	24	3505.447979	146.0603325	4228.418	65.02629	

B	22	1769.174577	80.41702622	629.8397	25.09661		
C	24	4027.960303	167.8316793	6848.263	82.75423		
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	94040.17097	2	47020.08548	11.75545	4.21E-05	3.133762	
Within Groups	267990.2991	67	3999.85521				
Total	362030.4701	69					
SD							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	65.64330625	24	22	13.19985	4.973033	YES
B	C	87.41465306	22	24	13.19985	6.622396	YES
C	A	21.77134681	24	24	12.90971	1.686432	NO

Anova: Single Factor - VSS						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	23	395200	17182.6087	22317411		
B	25	410761.2857	16430.45143	29784027		
C	24	402316.4573	16763.18572	17912093		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	6789354.327	2	3394677.164	0.144787	0.865469	3.129644
Within Groups	1617777816	69	23446055.3			
Total	1624567170	71				
NO SD						

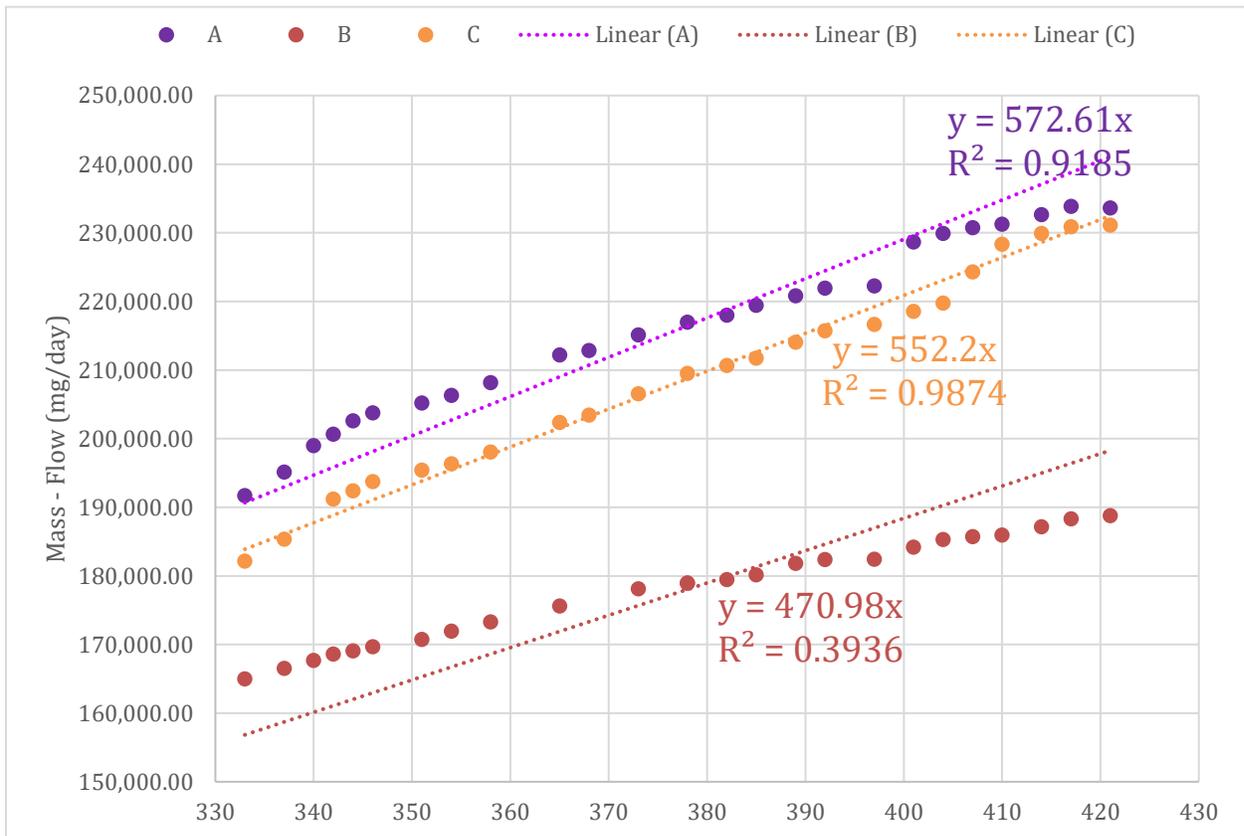
There IS NOT a difference						

Anova: Single Factor - FSS							
SUMMARY							
Groups	Count	Sum	Average	Variance			
A	26	106350	4090.384615	2504604			
B	24	95250	3968.75	1076481			
C	24	132341.1322	5514.21384	9436373			
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	35810985.13	2	17905492.57	4.176232	0.019288	3.125764	
Within Groups	304410737	71	4287475.169				
Total	340221722.1	73					
SD							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	121.6346154	26	24	414.4561	0.29348	NO
B	C	1545.46384	24	24	422.6639	3.656484	YES
C	A	1423.829224	24	26	414.4561	3.435416	YES

M – Permeate Calculations for Phase 2

Anova: Single Factor - Permeate - phase 2					
SUMMARY					
Groups	Count	Sum	Average	Variance	
A	24	1276.539	53.18913	1489.069	
B	24	718.1083	29.92118	198.0595	

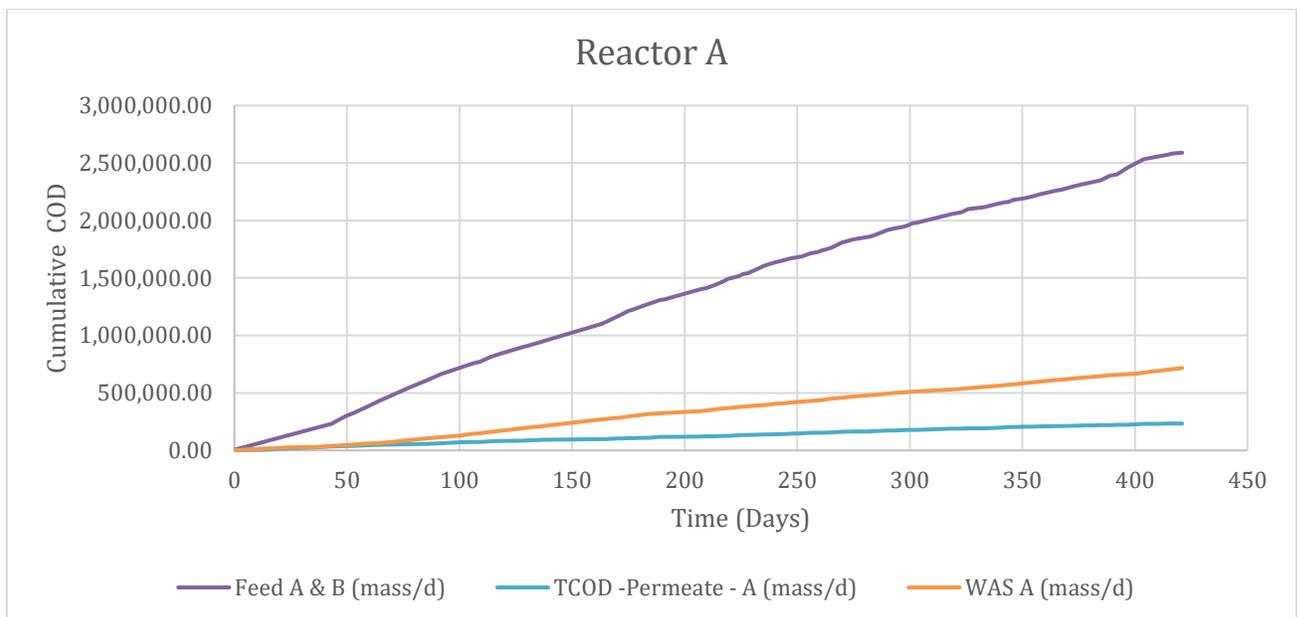
C	24	1414.198	58.92493	1345.479			
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	11324.11	2	5662.056	5.601176	0.00557	3.129644	
Within Groups	69749.97	69	1010.869				
Total	81074.08	71					
SD							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	23.26795	24	24	6.489957	3.585224	YES
B	C	29.00374	24	24	6.489957	4.469019	YES
C	A	5.735796	24	24	6.489957	0.883796	NO

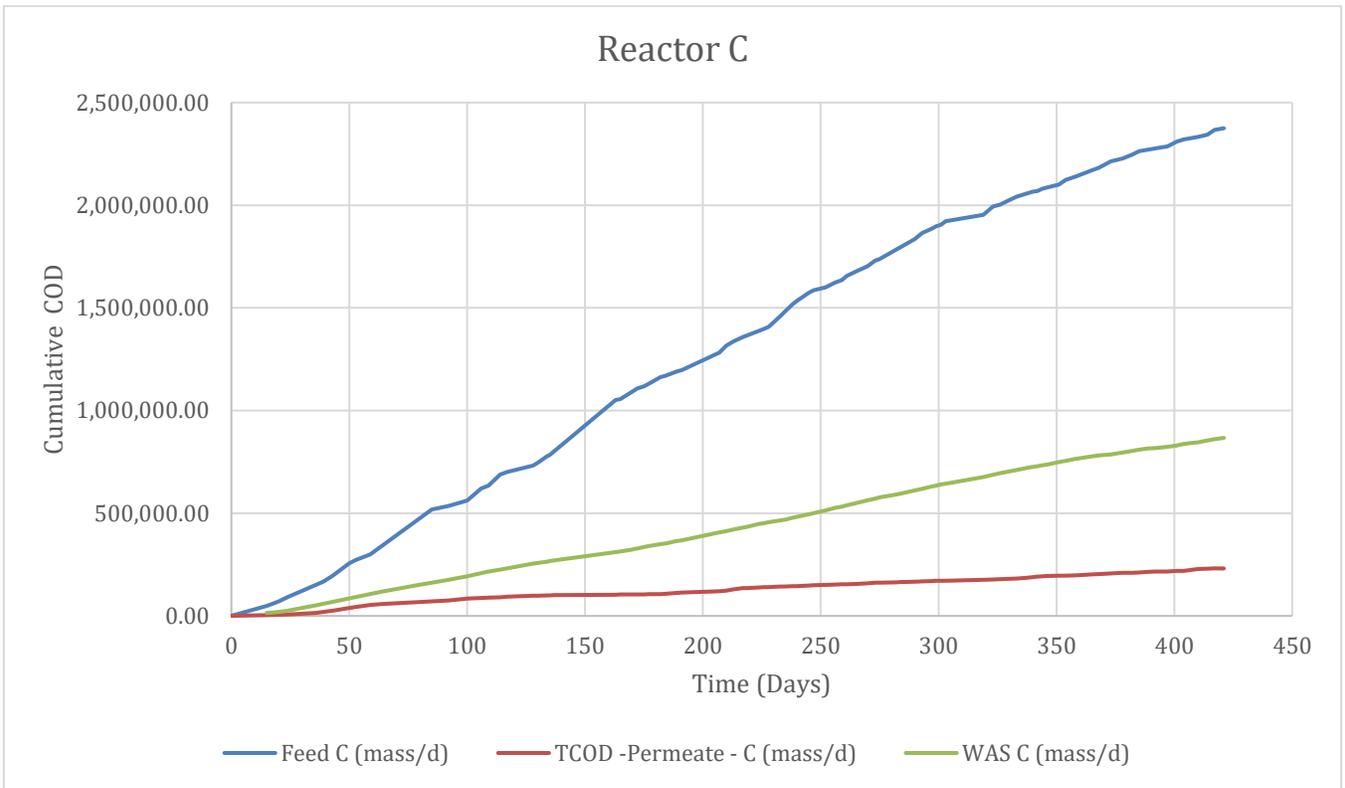
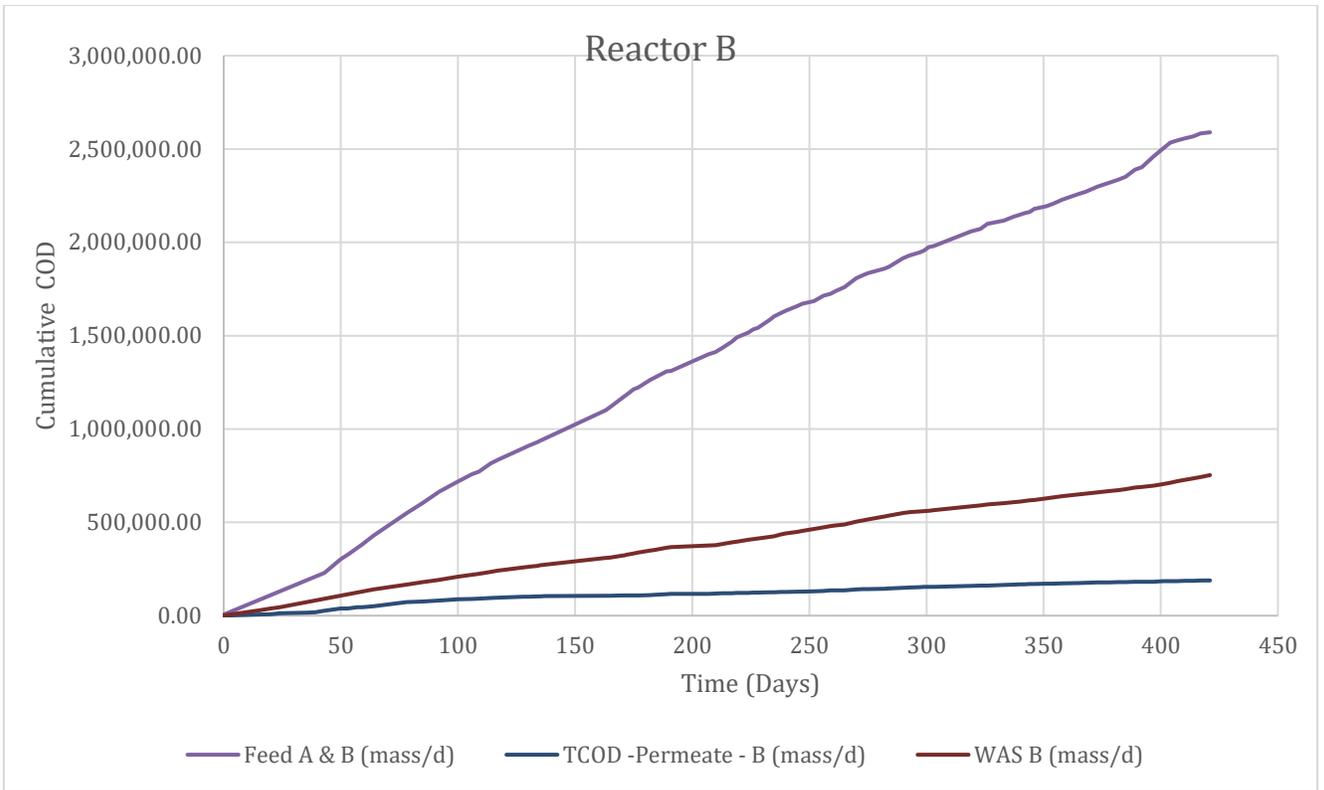


N – Effect of PAC on % removal

Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	11	987.7309	89.79371976	22.43191		
B	12	1100.413	91.70111176	17.04271		
C	11	956.672	86.97018482	34.25871		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	129.3902864	2	64.69514319	2.658554	0.085976899	3.304817
Within Groups	754.3760503	31	24.3347113			
Total	883.7663367	33				
NO SD						
There IS NOT a difference						

O – COD Balance for Phase 2





P – LCOCD Calculations for Phase 2

Humics

Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	7	34255	4893.5556	1668341.873		
B	7	27832	3975.9929	693475.1334		
C	7	35507	5072.4402	1912057.778		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	5E+06	2	2422137.5	1.700193103	0.210696846	3.5545571
Within Groups	3E+07	18	1424624.9			
Total	3E+07	20				
NO SD						

Bio-polymers - phase 2						
A	B	C				
18256	4893	18374				
9073	4808	12089				
11610	6810	12020				
6277	2316	4051				
8044	4634	12097				
8955	4668	10472				
	3155	8745				
Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	6	62214	10369.011	17914188.02		
B	7	31283	4469.0345	2033148.257		
C	7	77849	11121.281	18560047.16		

ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	182707172.7	2	91353586	7.286680182	0.005183093	3.5915306	
Within Groups	213130112.6	17	12537065				
Total	395837285.3	19					
SD							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	5900	6	7	1392.932205	4.2356527	YES
B	C	6652.2	7	7	1338.285975	4.9707211	YES
C	A	752.27	7	6	1392.932205	0.5400617	NO

Building Blocks - Phase 2					
A	B	C			
2718.62	1099.862069	2137.9			
1541.03	1576.896552	1675.9			
1579.45	1204.482759	1823			
1305	1089	1263			
4579.66	1882.758621	1857.7			
1710.34	1600.827586	7771.7			
2731	1323	2896			
Anova: Single Factor					
SUMMARY					
Groups	Count	Sum	Average	Variance	
A	7	16165	2309.2414	1331849.638	
B	7	9776.9	1396.6995	89234.63405	
C	7	19425	2774.9557	5104907.717	
ANOVA					

Source of Variation	SS	df	MS	F	P-value
Between Groups	6881495.833	2	3440747.9	1.581712599	0.232905059
Within Groups	39155951.93	18	2175330.7		
Total	46037447.77	20			
NO SD					
There IS NOT a difference					

JUST to be sure the LMWs were also tested

LMW Acids						
A	B	C				
6626.48	2972.068966	4296.8				
3536.55	4077.586207	3663.3				
4003.45	1426.206897	2305.2				
6884.41	5422.551724	7245				
5063.17	4383.448276	4881.7				
3944.48	3214.896552	6443				
3811.03	2407.724138	4685				
Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
6626.48	6	27243	4540.5172	1590162.302		
2972.07	6	20932	3488.7356	2076899.774		
4296.83	6	29223	4870.5172	3236628.086		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	6248929.938	2	3124465	1.357736904	0.287103608	3.6823203
Within Groups	34518450.81	15	2301230.1			
Total	40767380.75	17				
NO SD						
There IS NOT a difference						

LWM Neutreal						
A	B	C				
14454.6	231.7707664	1698.6				
223.253	2192.055354	321.55				
1660.92	289.1598201	6761.1				
0	0	0				
57756.2	742.7503116	669.57				
179.151	2878.171659	58515				
7502	148	15304				
Anova: Single Factor						
SUMMARY						
Groups	Count	Sum	Average	Variance		
A	7	81776	11682.273	441417494.9		
B	7	6482.1	926.0194	1299562.654		
C	7	83270	11895.714	452800638		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	550845751.7	2	275422876	0.922671469	0.415456036	3.5545571
Within Groups	5373106173	18	298505899			
Total	5923951925	20				
NO SD						
There IS NOT a difference						

Q –Cleaning frequency

Q1 - Significant difference in Cleaning frequency

Anova: Single Factor								
SUMMARY								
Groups	Count	Sum	Average	Variance				
A	45	141	3.133333	1.118182				
B	46	151	3.282609	2.029469				
C	16	164	10.25	5				
ANOVA								
Source of Variation	SS	df	MS	F	P-value	F crit		
Between Groups	675.1468	2	337.5734	162.8927	0	3.083706		
Within Groups	215.5261	104	2.072366					
Total	890.6729	106						
SD								
There IS a difference								
Tukey								
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?	
A	B	0.149275	45	46	0.213429	0.699414	NO	
B	C	6.967391	46	16	0.295444	23.58278	YES	
C	A	7.116667	16	45	0.29629	24.01926	YES	

Q2 - Normal Testing

A - phase 1		B - phase 1		C- phase 1	
Mean	3.133333	Mean	3.282609	Mean	10.25
Standard Error	0.157634	Standard Error	0.210045	Standard Error	0.559017
Median	3	Median	3	Median	10
Mode	3	Mode	3	Mode	10
Standard Deviation	1.057441	Standard Deviation	1.424594	Standard Deviation	2.236068
Sample Variance	1.118182	Sample Variance	2.029469	Sample Variance	5

Kurtosis	-0.26395		Kurtosis	0.518829		Kurtosis	0.062593
Skewness	-0.03594		Skewness	0.582848		Skewness	0.827984
Range	4		Range	6		Range	8
Minimum	1		Minimum	1		Minimum	7
Maximum	5		Maximum	7		Maximum	15
Sum	141		Sum	151		Sum	164
Count	45		Count	46		Count	16
Largest(1)	5		Largest(1)	7		Largest(1)	15
Smallest(1)	1		Smallest(1)	1		Smallest(1)	7
Confidence Level(95%)	0.31769		Confidence Level(95%)	0.423052		Confidence Level(95%)	1.191517
All are normal							

A – phase 2		B– phase 2		C– phase 2	
Mean	5.866667	Mean	6.428571	Mean	6.846154
Standard Error	0.400793	Standard Error	0.291157	Standard Error	0.355292
Median	7	Median	7	Median	7
Mode	7	Mode	7	Mode	7
Standard Deviation	1.552264	Standard Deviation	1.08941	Standard Deviation	1.281025
Sample Variance	2.409524	Sample Variance	1.186813	Sample Variance	1.641026
Kurtosis	-1.1696	Kurtosis	0.514918	Kurtosis	1.33699
Skewness	-0.53923	Skewness	-1.03692	Skewness	-0.79005
Range	5	Range	4	Range	5
Minimum	3	Minimum	4	Minimum	4
Maximum	8	Maximum	8	Maximum	9
Sum	88	Sum	90	Sum	89
Count	15	Count	14	Count	13
Largest(1)	8	Largest(1)	8	Largest(1)	9
Smallest(1)	3	Smallest(1)	4	Smallest(1)	4
Confidence Level(95%)	0.859615	Confidence Level(95%)	0.629006	Confidence Level(95%)	0.774116
All are normal					

R- Between phases comparison

R1 – Percentage Removal

Anova: Single Factor					
SUMMARY					
Groups	Count	Sum	Average	Variance	
NO PAC	38	3407.397	89.66835	52.97541	
0.5 PAC	11	987.7309	89.79372	22.43191	

1 PAC	12	1100.413	91.70111	17.04271		
2 PAC	11	956.672	86.97018	34.25871		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	129.6273	3	43.20911	1.08243	0.362493	2.739502
Within Groups	2714.466	68	39.91862			
Total	2844.093	71				
NO SD						
There IS NOT a difference						

R2 - Hypothesis testing

Model 1 - Sample

Permeate Cumulative Mass (mg)	Xp	Xrb	Xrc	Days	Interact. days.xp	Interact. days.xrb	Interact. days.xc
390.35	1	0	0	0	0	0	0
6,732.50	1	0	0	15	15	0	0
8,091.99	1	0	0	17	17	0	0
18,632.72	1	0	0	24	24	0	0
24,953.79	1	0	0	36	36	0	0
34,397.22	1	0	0	43	43	0	0
40,854.65	1	0	0	53	53	0	0
45,879.43	1	0	0	59	59	0	0
47,507.30	1	0	0	64	64	0	0
57,776.87	1	0	0	85	85	0	0

SUMMARY OUTPUT – MODEL 1							
Regression Statistics							

Multiple R	0.99							
R Square	0.98							
Adjusted R Square	0.98							
Standard Error	8561.95							
Observations	205.00							
ANOVA								
	df	SS	MS		F	Signifi- F		
Regression	7.00	8.26E+11	117975062564.87					1609.33
Residual	197.00	1.44E+10	73307064.58					
Total	204.00	8.40E+11						
	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95%	Upper 95%
Intercept	35498.48	13829.57	2.57	0.01	8225.47	62771.48	8225.47	62771.48
Xp	-33252.30	13813.82	-2.41	0.02	60494.24	-6010.36	60494.24	-6010.36
Xrb	20307.59	2921.44	6.95	0.00	14546.28	26068.91	14546.28	26068.91
Xrc	7155.26	3024.82	2.37	0.02	1190.08	13120.44	1190.08	13120.44
Days	474.01	36.95	12.83	0.00	401.14	546.88	401.14	546.88
Interact.days.xp	140.43	37.81	3.71	0.00	65.87	214.99	65.87	214.99
Interact.days.xrb	-145.92	11.30	-12.91	0.00	-168.20	-123.63	-168.20	-123.63
Interact.days.xc	-33.82	11.57	-2.92	0.00	-56.65	-11.00	-56.65	-11.00
	Slopes							
A1	614.45							
A2	474.01							
B1	468.53							
B2	328.10							
C1	580.62							
C2	440.19							

SUMMARY OUTPUT - Model 2				Second Model		Slope of A = B = C		
Multiple R	0.979136103							
R Square	0.958707508							
Adjusted R Square	0.958091203							
Standard Error	13138.51108							
Observations	205							

ANOVA								
	df	SS	MS	F	Significance F			
Regression	3	8.06E+11	2.69E+11	1555.571	0			
Residual	201	3.47E+10	1.73E+08					
Total	204	8.40E+11						
	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95%	Upper 95%
Intercept	44223.25	21042.63	2.10	0.04	2730.62	85715.88	2730.62	85715.88
Xp	-32011.52	21193.48	-1.51	0.13	-73801.60	9778.55	-73801.60	9778.55
Days	414.96	55.68	7.45	0.00	305.18	524.75	305.18	524.75
Interact.days.xp	133.97	58.01	2.31	0.02	19.59	248.35	19.59	248.35
	Slope							
Phase 1 (A ,B,C)	548.9328447							
Phase 2 (A ,B,C)	414.9635357							
http://www.statisticslectures.com/tables/ftable/								
IS it different								
F (calculated)	69.07664203							
f (From table)	2.441							
69 >> 2.447	SO M1 is better							
	So A ,B and C are significantly different							

S – TCOD WAS Phase 1

WAS A	WAS B	WAS C
8897.63633	8,791.49	7,264.87
8012.55533	13,737.54	7,607.33
6907.45032	15,098.58	10,301.24
6904.738	13,386.06	17,239.46
2472.054	17,979.87	17,885.22
9556.866	13,809.41	15,459.41
5746.9764	14,125.34	15,636.40
14821.91	12,904.39	13,529.90
13875.546	16102.627	17296.006
12928.4487	12651.756	19127.7583
13083.64	12009.888	16954.152
19131.7387	15572.541	14807.745
11847.648	18777.7593	17155.1153
17615.746	10851.3967	15020.784
18796.2827	12533.427	14966.3727
17281.448	14358.803	12266.706
18484.9753	10590.848	13589.7443
12122.7613	10394.441	11952.7847
13079.825	14692.148	12944.2373
14740	18580.1247	14025.176
16777.3987	17217.696	15348.5807
12385.2833	19728.3893	14718.567
16239.104	17442.01	17823.2333
15750.514	14888.8017	17104.246
21073.2053	12562.98	14852.0067
19886.778	18283.382	14595.108
16393.3083	12448.3	16524.0487
12424.996	3853.724	15920.768
9989.382	16317.4533	17831.7213
11111.4813	17518.8733	16709.824
8030.296	10910.72	18363.3733
14687.712	15255.2407	17317.4013
17566.317	12090.6173	16375.52
16456.948	16633.9688	21398.74
13213.28	12859.912	15001.1347
14076.9813	13680.03	17330.4787
14339.332	23472.6833	12543.3467
11165.7307	20181.34	13625.625

11425.785		21405.7083
16178.875		19364.6733
14648.4233		

Anova: Single Factor								
SUMMARY								
Groups	Count	Sum	Average	Variance				
WAS A	41	550129.377	13417.7897	17151490.4				
WAS B	38	552294.556	14534.0673	12950731.4				
WAS C	40	619184.504	15479.6126	9181190.59				
ANOVA								
Source of Variation	SS	df	MS	F	P-value	F crit		
Between Groups	86320819	2	43160409.5	3.28667845	0.04088752	3.07		
Within Groups	1523303110	116	13131923.4					
Total	1609623929	118						
SD								
There IS a difference								
Tukey								
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?	
A	B	1116.27758	41	38	577.004222	1.93	NO	
B	C	945.545333	38	40	580.463183	1.63	NO	
C	A	2061.82292	40	41	569.468567	3.62	YES	

T Test: Two-Sample Assuming Unequal Variances		
	WAS A + WAS B	WAS C
Mean	13954.7333	15479.6126
Variance	15253989.5	9181190.59
Observations	79	40
Hypothesized Mean Difference	0	

df	97	
t Stat	-2.34563991	
P(T<=t) one-tail	0.01051529	
t Critical one-tail	1.66071455	
P(T<=t) two-tail	0.02103058	
t Critical two-tail	1.98472314	
There is a difference P<0.05		

T – LCOCD t –Test

T1 – Humics

Humics	
NO PAC	PAC
5891.448	5446.364
5661.586	4009.506
9297.655	4668.457
11220.69	3249.076
8545.655	6940.746
4354.207	3979.125
4222.759	5961.617
5728	3287.746
4181	3541.048
2272.872	3319.116
3125.199	3274.155
4688.552	5195.181
4594.759	4226.38
4970.483	4988.324
4789.31	5532.31
6357.724	5473.622
4341.517	3460.368
4502	3049.356
4458	5655.948
4992.92	5254.382
1059.073	7081.094
4265.655	
4409.103	
4574.069	
6282.621	
4861.793	

4712	
5432	
9898.58	
1063.533	
1063.533	

T Test: Two-Sample Assuming Unequal Variances		
	NO PAC	PAC
Mean	5026.375	4647.33
Variance	5358995	1524376
Observations	31	21
Hypothesized Mean Difference	0	
df	47	
t Stat	0.765068	
P(T<=t) one-tail	0.224028	
t Critical one-tail	1.677927	
P(T<=t) two-tail	0.448056	
t Critical two-tail	2.01174	
There is NO Sig dif		

T2 - Biopolymers

T Test: Two-Sample Assuming Unequal Variances		
	NO PAC	1.0 PAC
Mean	11486.3	4469.034
Variance	59221029	2033148
Observations	32	7
Hypothesized Mean Difference	0	
df	36	
t Stat	4.795662	
P(T<=t) one-tail	1.4E-05	
t Critical one-tail	1.688298	
P(T<=t) two-tail	2.8E-05	
t Critical two-tail	2.028094	

There is a SD		
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T3 - Building Block

T Test: Two-Sample Assuming Unequal Variances		
	No PAC	PAC
Mean	2402.927	2160.299
Variance	5598482	2301872
Observations	32	21
Hypothesized Mean Difference	0	
df	50	
t Stat	0.45483	
P(T<=t) one-tail	0.3256	
t Critical one-tail	1.675905	
P(T<=t) two-tail	0.6512	
t Critical two-tail	2.008559	
There is NO Sig dif		

U – T- test for Cleaning frequency

T Test: Two-Sample Assuming Equal Variances		
	NO PAC	PAC
Mean	3.208791	6.357143
Variance	1.567033	1.844948
Observations	91	42
Pooled Variance	1.654014	
Hypothesized Mean Difference	0	
df	131	
t Stat	-13.123	
P(T<=t) one-tail	6.15E-26	
t Critical one-tail	1.656569	
P(T<=t) two-tail	1.23E-25	
t Critical two-tail	1.978238	
There is a SD		

V- TMP Phase 2

A	B	C
7.25	6.39	8.97
6.24	7.22	8.67
6.13	7.25	7.79
4.84	7.85	9.35
6.06	7.38	7.92
6.84	5.72	8.22
4.91	8.23	6.34
4.91	6.90	8.76
6.34	7.09	8.18
5.87	6.63	8.16

Anova: Single Factor							
SUMMARY							
Groups	Count	Sum	Average	Variance			
A	10	59.371829	5.937183	0.682749			
B	10	70.655217	7.065522	0.511255			
C	10	82.367691	8.236769	0.680998			
ANOVA							
Source of Variation	SS	df	MS	F	P-value	F crit	
Between Groups	26.443552	2	13.22178	21.15482	2.97E-06	3.354131	
Within Groups	16.875021	27	0.625001				
Total	43.318573	29					
SD							
There IS a difference							
Tukey							
		Difference	n(group 1)	n(group 1)	SE	q	Sig Diff ?
A	B	1.1283388	10	10	0.25	4.513352	YES
B	C	1.1712474	10	10	0.25	4.684987	YES
C	A	2.2995862	10	10	0.25	9.198339	YES

To work out critical q							
df	27						
# of group	3						
USE 30	3.49						

W- Apparatus

PAC Properties

Related Categories

Activated Charcoal, Activated and Mesoporous Carbon, Adsorbents, Filter Aids and Drying Agents, Alternative Energy, Chemical Synthesis, [More...](#)

biological source	wood
InChI Key	OKTJSMMVPCPJKN-UHFFFAOYSA-N
form	powder
quality	chemically activated
mfr. no.	Norit CA1
greener alternative product characteristics	Design for Energy Efficiency Learn more about the Principles of Green Chemistry.
ign. residue	≤4.0%
loss	≤16% loss on drying

[Show Fewer Properties](#)

This choose can set the width

Once translation bundle is plugged in need to wrap this to use key

Description

changed H5 to H4 for 2540

Application

Activated carbons may be used in:

- Separating gas mixtures, such as; hydrogen-hydrocarbons,
- Gas drying;
- Selective gas adsorption
- Gas storage

Activated charcoal Norit may be used in recovery of lithium for recycling lithium-ion batteries. It may be used to study the adsorption isotherm of 2,4,6-trichlorophenol (TCP). 6 Activated carbon may be used for ultrapurification of water, such as removal of anti-anxiety drug diazepam (valium) from spiked

wastewater.

General description

Activated carbon (AC) is a three dimensional highly microporous structure with high surface area. AC can be prepared from various carbon precursors, such as lignite, peat, wood, coal etc. AC powder is popularly used in adsorption or as catalyst support. The adsorption capability of activated carbon is determined by its pore size distribution, purity and degree of carbonization, surface chemistry. Method of activation also influences the final properties of the product. Activated carbon prepared from lignite are hard textured with small micropore volume and high pore size distribution.4 It is used as a carrier for catalysts and possess good filtering properties.

Masterflex L/S Digital Pump System with Easy-Load® 3 Pump Head, 600 rpm, 115/230 VAC Masterflex – Item # HV-77921-65

Specifications & Description

Pump Series	L/S
Control Type	Variable-Speed Digital
Min RPM	0.1
Max RPM	600
Number of Channels	1
Max Flow Rate Per Channel (mL/min)	480
Min Flow Rate Per Channel (mL/min)	0.08
Drive	07522-20 (L/S® Brushless Digital Dispensing Drive)
Pump Head	77800-60 (L/S® Easy-Load 3 Pump Head)
Tubing	06434-16, 10-ft (C-FLEX® Ultra, L/S® 16)
Tubing sizes accepted	L/S® 13, 14, 16, 25, 17, 18
Drive IP rating	IP 31
Power (VAC)	90 to 260
Power (amps)	2.2A at 115v; 1.1A at 230v
Power (Hz)	50/60
Width (in)	8.3
Height (in)	8.5
Length (in)	13.88
Width (cm)	21.08
Height (cm)	21.59
Length (cm)	35.26
Description	L/S Digital Pump System with Easy-Load 3 Pump Head, 600 RPM, 115/230V

KNF LABOPORT® N 810 FTP Diaphragm Vacuum Pump, 10 L/min, 75 torr; 115 VAC

Specifications & Description

Wetted Parts PTFE, FFPM
Free-Air Capacity (CFM) 0.35
Free-Air Capacity (LPM) 10
Free-Air Capacity (LPH) 600
Max Flow Rate (CFM) 0.35
Max Flow Rate (Liters/min) 10
Max Pressure (PSI) 14.5
Max Pressure (bar) 1
Max Vacuum (in Hg) 27
Max Vacuum (Continuous) (in Hg) 27
Max Vacuum (Intermittent) (in Hg) 27
Max Vacuum (mbar) 100
Max Vacuum (mm Hg) 75
Max Vacuum (torr) 75
Max Vacuum (micron) 100000
Max Vacuum (in H₂O) 36.4
Port Size G1/8", Includes 10mm hose barb
Max Temperature (° F) 104
Max Temperature (° C) 40
Max Temperature (Media) (° F) 104
Max Temperature (Media) (° C) 40
Max Temperature (Ambient) (° F) 104
Max Temperature (Ambient) (° C) 40
Duty Cycle Continuous
Power (VAC) 115
Power (amps) 1.3
Phase 1
Power (Hz) 60
Motor Type TEFC
Stages One
Shipping Weight (kg) 6.9
Shipping Weight (lbs) 15.2
Height (in) 7.4
Width (in) 5.7
Length (in) 10.1
Height (cm) 18.7
Width (cm) 14.6
Length (cm) 25.6
Height (mm) 187
Width (mm) 146
Length (mm) 256
Description Diaphragm Vacuum Pump, 10 L/min, 75 torr; 115 VAC

**Thin Film Pressure Sensor with 100 mV Output Excellent Long
Term Stability Item# PX602-015GV
SPECIFICATIONS**

Excitation: 10 Vdc (5 to 10 Vdc limits)

Output: 0 to 100 mV @ 10 Vdc Sensitivity: 10 mV/V

Input Impedance: 1500 Ω Output Impedance: 100 Ω Insulation Resistance: 100 M Ω
@50 Vdc

Accuracy: $\pm 0.4\%$ BFSL Hysteresis: $\pm 0.2\%$ Repeatability: $\pm 0.05\%$ Stability: $\pm 1\%$ /year

Zero Balance: $\pm 1\%$ Durability: 100 million cycles Operating Temp: -48 to 91°C (-55
to 195°F)

Compensated Temp: -29 to 82°C (-20 to 180°F)

Thermal Zero Effect: $\pm 0.07\%$ full scale/°C

Thermal Span Effect: $\pm 0.07\%$ full scale/°C

Proof Pressure:

15 to 2000 psi = 200% 3000 to 5000 psi = 150% 7500 to 20,000 psi = 120%

Burst Pressure:

15 to 2000 psi = 800% 3000 to 20,000 psi = 500%

Gages: Thin film polysilicon Diaphragm: 17-4 PH stainless steel Case: 300 Series
stainless steel Pressure Connection:

15 to 10,000 psi: 1/4 NPT

15,000 and 20,000 psi: 9/16-18 UNF Aminco fitting

Electrical Connection: 0.9 m (36") braided-shield PVC cable or connector Weight: 71
g (2.5 oz.) without cable Response time: 5 ms

Construction: Sealed units (except PX602 \leq 500 psi is vented to room)