An empirical analysis of cleaning frequency impacts on the process performance of a lab scale gas-sparged anmbr treating swine wastewater

by

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Abstract

For 9,000 years, pigs have been intertwined with human societies, as our population rose from 10-20 million to 8 billion today. Global swine rearing has expanded alongside human development, straining the Earth's waste management capacity. Climate change-induced disaster and the eminent crossing of planetary boundaries in phosphorus and nitrogen utilization emphasize the urgent need for efficient waste management. Swine, primarily and increasingly raised in concentrated animal feedlot systems (CAFOs), generate excessive manure, leading to as much as 170 million tons CO_2e emissions and significant financial losses in the form of wasted nitrogen and phosphorus, loss of ecosystem services, environmental remediation, and uncaptured energy potential. Improved swine manure management is crucial for sustainable development.

The Anaerobic Membrane Bioreactor (AnMBR) holds potential as a treatment and resource recovery solution, aiming for net-zero or energy-negative operation. However, membrane fouling challenges treatment efficiency and cost-effectiveness. To date, there have been only a few lab and bench scale studies on swine wastewater treatment with AnMBRs, most of which use a synthetic or controlled swine waste influent (by dilution and mixing with synthetic solutions) and focus on acclimation of the microbial community and COD conversion to methane. While these studies have implemented various fouling management technologies, further research into energy optimization of AnMBRs treating high-strength waste streams like swine manure is warranted given the technology's novelty. This study seeks to explore optimization options for a completely submerged AnMBR configuration using common fouling management techniques and real, highly variable swine wastewater influent.

The completely submerged, continuously gas sparged 114.7 L AnMBR was operated in two discrete phases, both targeting distinct objectives. Phase I aimed to minimize chemical

cleaning footprint for fouling control while preserving membrane performance and permeate flux. The volumes of citric acid and bleach used for CIP maintenance were limited to 250 mL per 10-minute cleaning cycle compared to the manufacturer protocol of 15 L for 5 hours, and the cleaning frequency was varied between 0.37 and 2.5 times per week over 244 days (manufacturer recommendation of 2.0 times per week). 60-80% COD removal was observed during this period, which is low for such systems. The highest average permeability recovery (15.2%) and recovery efficiency (23.8%/%) were observed during the Case 2. However, no correlation was established between cleaning frequency and permeability recovery. The average overall flux was maintained at an acceptable 10.2 LMH, supporting that cleaning schedule may have little impact on gas sparged AnMBR efficiency and can likely be significantly reduced compared to conservative manufacturer recommendations.

Phase II, spanning 114 days, used higher strength waste and sustained a more consistent operation schedule under a constant but reduced cleaning schedule, and demonstrated consistent average COD and BOD removal (87.9% and 90.7% respectively) comparable to high-performance anaerobic and facultative lagoon systems and low to mid performance AnMBRs, with an average HRT of 7.6 days, significantly shorter than traditional AD systems (20-75 days). Effluent turbidity stayed within range for indirect reuse (average: 0.44 NTU), indicating superior solids removal. Between days 52 and 103, an estimated 45.6% of the COD input was converted to methane in the headspace biogas, which is in line with expectations for these systems (30-75%). The average methane concentration in the biogas was 72.6%, with a peak of 81.9%, showing promise for upgradation to pipeline injection standard, and further supporting that gas sparging for fouling mitigation has the added benefit of enriching the biogas methane content by leveraging the greater solubility of CO_2 compared to CH_4. Due to the consistent operation, gas greater permeability recovery (average +2.44% pts) than chemically enhanced backpulse (CEB), which further supports that gas sparging with passive batch recovery (PBR) may be a preferred method of fouling management in swine AnMBRs to CEB.

An initial modeling framework to model fouling behavior in continuously gas sparged, submerged AnMBRs was developed that successfully described 67% of the observed data and demonstrated an R^2 0.8892 when the real data was plotted against the predictions. This indicates there is promise for

In summary, the findings of this study agree with past observations from AnMBR operation and support that the high performance and energy recovery potential of such systems can be extended to swine wastewater, a prevalent waste stream and polluter. The resilience of superior COD and BOD_5 removal to variable organic loading rates increases confidence in the capability of AnMBRs to manage high strength wastewaters. It also supports that gas sparging as a fouling management option has added benefit by significantly reducing chemical cleaning footprint and enriching methane content of biogas.

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Dedication

I would like to Dedicate this Thesis to Antonio Landa, Miguel Contreras and the people of El Picacho Honduras for teaching me the importance of water, the worth of engineering and the pride to be found in good work; Dustin Burton, Chase Kohler and the communities of the greater KC metro for my cradle as a young engineer; to Aaron Smith, Janey Camp and Prathap Parameswaran for my spark as a student and researcher; to the Civil engineering department at K-State for this opportunity, and every Music and Art teacher who has ever empowered me with the tools to make life beautiful by giving me some of theirs, particularly Allison Ross, Katie Benyo and Ms. Gibbs.

Above all, I would like to dedicate this work to God Almighty and the Methanogens put on this earth by God's hand and that lived and died in the name of this research. They teach us what it means to pursue life tenaciously and give as much as we can back. We would not be here without them, and I am honored to be a part of their story.

Chapter 1 - Introduction

Waste is no longer a designation we can afford to assign casually. The challenges of the twenty-first century make it increasingly clear that developed nations must pursue greater circularity in their economies and take advantage of opportunities to recover valuable resources from abundant waste streams[1], [2], [3]. The benefit of doing so compounds when the resources recovered would otherwise be released to the environment as damaging pollutants[4], [5]. This is the case with swine manure, which is responsible for costly releases of nutrients leading to water quality degradation and emissions of methane, nitrates, and carbon-dioxide that contribute to global warming[1], [6]. It has been determined in other studies that swine manure management accounts for approximately 23% of greenhouse gas emissions (GHGe) related to pork production, as much as 170 million annual tons CO_2e globally with China as the largest producer, while a report by the EPA puts the figure for US swine manure management alone at 20 million tons CO_2e[7], [8]. For England and Wales, it is reported that eutrophication due to agricultural N and P could account for as much as $35 million in losses and remediation costs, while Smith et. al reports that the value of nutrients N, P and Potassium (K) in livestock manures could amount to $300 million annually, with swine manure accounting for about 11% of the nation's manure production[9], [10], [11], [12]. By a U.S. Department of Energy estimate, the US manure production in dry tons from swine and dairy operations over 2000 and 500 heads respectively is at least double that of the UK's[1], [13]. In addition to China, the US, and the European Union, Brazil and Russia are top producers [14]. Clearly, for many nations around the world, recovery of resources from swine and other livestock manures holds significant value.

Traditional and current industry practice falls short of realizing the waste stream's potential. U.S. swine manure management is dominated by deep pit and lagoon storage, from which effluent slurries and nutrient loaded waters are often land applied for nutrient recovery and waste disposal. Uncovered lagoons, land spreading and to a lesser extent slurry injection facilitate nutrient and carbon release to the environment as GHG (N_2O, CO_2, CH_4) and water body pollution through runoff, erosion and leachate[6], [15]. The most prevalent systems in the U.S. for waste carbon valorization to biogas are covered anaerobic digestion lagoons, though complete mix systems have seen significant adoption as well [13], [16], [17]. However, the EPA estimates all AD systems in use as of 2021 accounted for only about 3% of potential GHGe reduction[13]. Meanwhile, even covered lagoons and other, more complex AD systems in use typically address only carbon valorization to energy and do not enhance nutrient recovery, water quality and water reuse, nor provide a pathway for commercialization of volatile fatty acids produced as biproducts of AD [16], [18], [19]. Diversion of resources contained within swine waste from environmental fates as pollutants back to agricultural, municipal and industrial use is a vital step in achieving a circular economy and sustainable future.

Anaerobic membrane bioreactors (AnMBRs) represent an innovation in wastewater treatment that combines the biological treatment and byproduct valorization of an anaerobic digester with the physical separation technology of membrane filtration. While aerobic MBRs have been studied at scale, and the ability of AnMBRs to effectively treat various waste streams has been supported, there exists a gap in the understanding of fouling mechanics and management of AnMBRs treating high strength wastes such as swine manure. Fouling management and membrane life cycle are two of the primary factors determining the cost effectiveness of AnMBR implementation[20], [21], [22], [23], [24]. Thus, more study on the effects of fouling on process performance and on the optimization of common fouling control strategies is warranted.

To operate and understand data extracted from the AnMBR platform, it is necessary to have a grasp of its constituent subsystems; anaerobic digestion and microbial communities, critical flux in theory and in application, fouling pathways and behavior, current fouling control techniques, and operational parameters and responses for past and existing systems. These will be discussed in the following sections of this chapter.

1.A. Anaerobic Digestion

Anaerobic digestion is carried out by microbes in the absence of oxygen. Oxygen, being the most energetic electron acceptor widely used by biologic electron transport systems, allows aerobic microorganisms to develop metabolisms that are, in many ways, not beneficial for wastewater treatment. Though aerobes have high rates of substrate utilization allowing rapid treatment, the energy required to provide oxygen for their metabolisms is large, and the amount of energy they are able to invest in their own biomass can prove problematic. As a result, most of the energy stored in the waste influent is used by the microbes, returning little value to the operators and human society other than the treated water.

Anaerobes, on the other hand, are used to working with less. Methanogens in particular are admirable, if long-suffering, allies for sustainable wastewater treatment operators. Methanogens are obligate anaerobic archaea that typically use hydrogen and carbon dioxide, and simple organic compounds such as acetate and methanol to extract and store energy by synthesis of methane, which can then be recovered in a treatment process to be used as energy for human society.

Table 1. Comparison of Free Energies Between Aerobic and Anaerobic Redox Reactions

	Oxidation		ΔG° (kJ/mol)
Propionate*	Aerobic	$C_3H_5OO^{-}+(7/2)O_2 \rightarrow 2CO_2+HCO_3^{-}+2H_2O$	-661.0
	Anaerobic*	$C_3H_5OO^{-}+(1/2)H_2O \rightarrow CH_3OO^{-}+(1/4)CO_2+(3/4)CH_4$	-28.0
Acetate*	Aerobic	$CH_3COO^{-}+3H_2O+2O_2 \rightarrow CO_2+HCO_3^{-}+ 4H_2O$	-848.4
	Anaerobic*	$CH_3COO^{-}+H^{+} \rightarrow CO_2+CH_4$	-75.8
Methanol	Aerobic	$CH_3OH+H_2O+(3/2)O_2 \rightarrow CO_2+ 3H_2O$	-693.9
	Anaerobic	$CH_3OH \rightarrow (1/4)HCO_3^{-} +(1/4)H_2O+(1/4)H^{+}+(3/4)CH_4$	-78.6
Hydrogen	Aerobic	$H_2+(1/2)O_2 \rightarrow H_2O$	-237.2
	Anaerobic	$H_2+(1/4)CO_2 \rightarrow (1/2)H_2O^{-}+(1/4)CH4$	-32.7

* These equations are carried out by two different microbial groups as part of syntrophic metabolism with each other with Hydrogen as the intermediate

As can be seen from Table 1, aerobes access far more substrate energy to invest in growth and maintenance than methanogens and much of the difference is conserved in synthesized methane. However, also illustrated by Table 1 is the reliance of methanogens (specifically hydrogenetrophs) on syntrophic relationships. Hydrogenotrophic methanogens scavenge hydrogen and CO_2 produced by fermentative bacteria in a process called reverse acetogenesis that is syntrophic in nature, while acetoclastic archaea convert the acetate from secondary fermentation to methane.

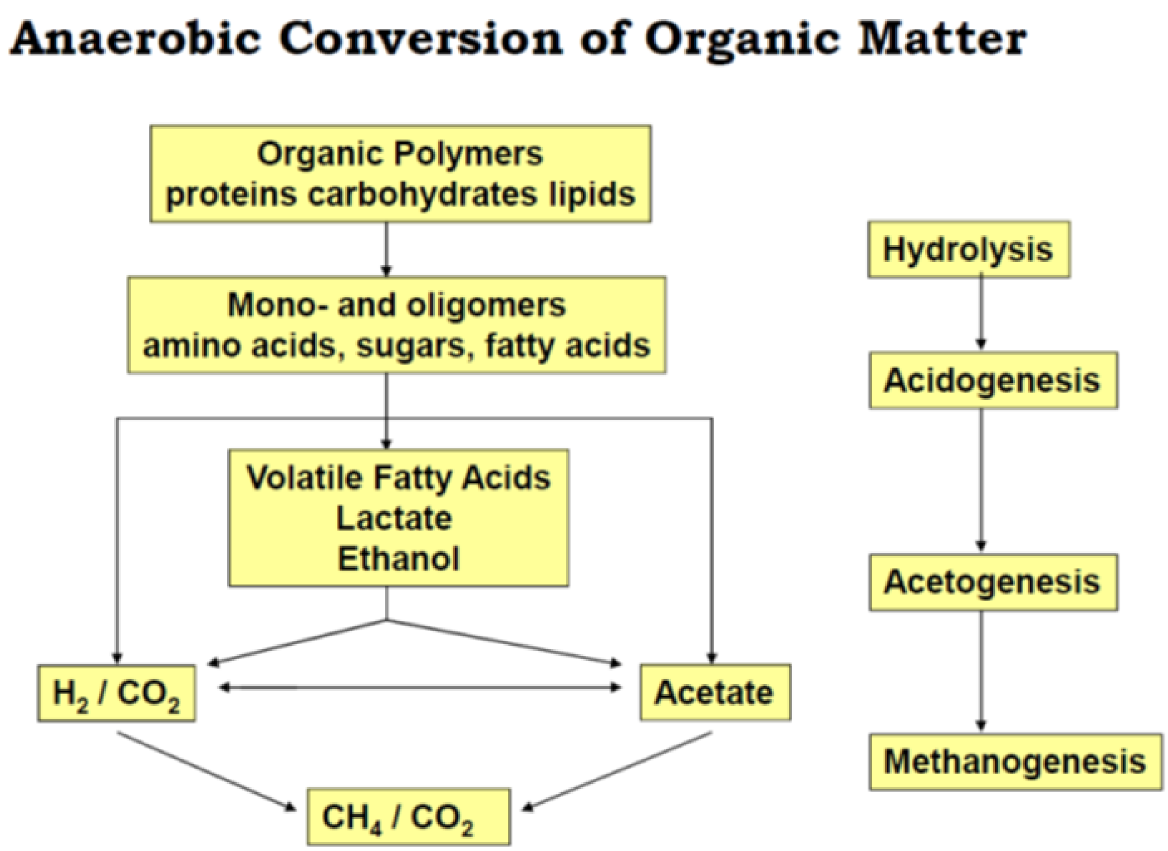

Figure 1. Overview of Anaerobic Digestion Food Web

Methanogenic metabolisms and their interactions with fermentative bacteria have been detailed elsewhere, but it is important to note that methanogenic groups can be sensitive to changes in pH, reduction potential of their environment, concentrations of ammonia, presence of oxygen and other toxicants as well as rare nutrients such as nickel used in their enzymatic processes[25], [26], [27]. Their success can also be limited by competition from other more energetic microbes; and the lack of cooperative fermenters. What methanogens, if any, can thrive to perform their desired roll of treatment and methane synthesis in an anaerobic bioreactor will depend on operational parameters such as the substrate availability and waste stream characteristics, SRT, temperature, pH, hydraulic conditions, and availability of surface to grow on in the reactor. methanogenic groups typically found in anaerobic digesters are:

- *Methanoculleus* belong to an order of Hydrogenotrophs [25], [28].
- *Methanosarcina* belong to a diverse order of methanogens with a wide range of metabolic functions including methylotrophic and acetoclastic subsistence. They are known to be able to carry out electrogenic processes [25], [28].
- *Methanobrevibacter* belong to an order of Hydrogenotrophs[25], [28].
- *Methanosaeta:*Acetoclastic methanogens found in Anaerobic Digester treating municipal and swine wastewater [25], [28].

What species dominate within the reactor and their places within the microbial community will also have an impact on the EPS generated and thus the fouling character of the system. Iron is an important micronutrient for the enzymatic processes of hydrogenotrophs and it is well documented that many microbes form chelates with iron within their extracellular matrix for storage. Similarly, it is known that syntrophic microbes tend to form structural connections to each other to enable electron transfer across their linked metabolisms.

Anaerobic digestion is growing in popularity for managing agricultural, food and pharmaceutical process wastes, and municipal wastewater treatment wastes. First adopted as a modern technology in India in 1859, there are between 1200 and 2400 AD systems in operation in the USA responsible for as much as 14 GWh of biogas-produced electricity[16], [29], [30]. In China, where efforts have begun to consolidate dispersed, small to mid-size digesters, there are likely nearer to 30,000 digesters in operation[31]. The numbers for Europe are greater, with Germany alone employing roughly 9,700 digesters and capacity for 32.5 TWh of electricity generation[18], [32], [33]. While the energy producing benefits of AD has been recognized globally, AnMBRs promise the ability to couple those benefits with superior water resource recovery, biogas quality, and fatty acid synthesis.

1.B. Critical Flux & Permeability

Flux in the context of membrane filtration refers to the rate at which a volume of filtered solution can be said to transition the bulk mixture across the membrane surface. It is a vital parameter for any membrane filtration process and has a natural limit in most processes known as the critical flux.

Building on the work of other membrane scientists of the 1980s, Field et al established in 1995 the definition for the concept of critical flux in membrane operation as the flux at which a solution filtrate flux versus driving transmembrane pressure (TMP) response diverges from that of pure water in a decreasing fashion[34], [35]. In practice, and particularly with biologically active, high solids reactor liquor filtration, this definition is of little importance as filtrate flux immediately diverges from pure water flux for TMPs in the range of realistic operation (ie; fluxes and corresponding TMPs necessary to achieve HRTs of 10 days or lower). Bacchin et al recodified the unruly tangle of terms and usages of terms that built on the concept of critical flux into Strong Form Critical Flux (Field’s original definition), Weak Form Critical Flux (a more useful definition for wastewater research), a threshold of reversibility critical flux, and a Sustainable Flux (a useful definition for membrane operators)[35]. Unless otherwise specified, usage of the term critical flux in this paper should be taken to mean weak form critical flux as defined by Bacchin, which is to say, it assumes rapid fouling and or antecedent fouling at the onset of filtration and refers to the point (TMP, Flux) where the behavior of TMP and flux response curve becomes nonlinear. Generally, the goal of both operator and optimizer is to reach a sustainable flux that allows operation within safe TMP bounds for an economically feasible time within the context of other operational parameters. Thus, critical flux on its own is not a concept that will be used extensively in this study; it is nonetheless vital for understanding of fouling.

In general, mass flux of particles in the colloidal range toward the membrane center, can be conceptualized mathematically as

$$N = J * C - D\frac{dC}{dy} + p(\varsigma) + q(\tau) \text{ [35]}$$

Where: *N* is the mass flux

J is the liquid flux

C is the bulk concentration

D is the dispersion term

ζ represents the electrokinetic repulsion or attraction of the particles to the membrane surface.

τ is the local hydraulic effect such as shear and filtration polarization on the particle behavior

Critical flux is directly related to permeability in the sense that permeability if the magnitude of flux achieved per unit of driving pressure. Permeability is considered to be a property of the membranes when it often combines both the membrane permeability and the mixture filterability. Nonetheless, a characteristic permeability of a membrane is often defined in a particular use at a particular pressure.

1.C. Fouling Pathways & Progression

Fouling is a process in membrane filtration related to critical flux whereby the effective area through which permeation is possible decreases and the resistance to flow increases due to the accumulation of material in the pores and on the membrane surface. Fouling is divided in three ways, by stage, by pathway, and by reversibility. A review of these categories, their uses and formulations will be helpful for clarity. In brief, fouling, or the resistance to permeation contributed by foulants, typically occurs in three stages: initial, rapid fouling, followed by an extended period of slow, stable, and roughly linear increase from which a parameter termed the fouling index (FI) is determined, and then a terminal fouling stage where fouling rate increases rapidly as the limiting flux becomes smaller due to deposition and compaction of material blocking the membranes [36], [37]. The three pathways most typically identified as contributors to fouling are:

- Cake formation, further classified as colloidal/organic and particulate fouling, with compaction in later stage of fouling termed as gel formation.
- Pore constriction, also called inorganic fouling or scaling, from precipitation of salts, though metalorganic complexes and other, internal pore blockage may play a large role in some cases.
- Biofilm formation, also called biofouling, referring to the blockages and increased structural stability of other fouling components created by microbial communities and their extracellular matrix.

[36], [37], [38], [39], [40], [41].

All three pathways are of importance in swine wastewater treatment. Once critical flux has been reached, by definition, the driving force toward the membrane is great enough to overcome charge resistances and diffusive action of solutes and fouling is presumed or observed to take place. In the case of completely submerged membrane bioreactors, fouling can occur without this driving force by adsorption and biofilm formation.

The final way fouling is categorized is by reversibility, where fouling processes and fouling states are referred to as reversible or irreversible. There are further classifications, but in general the usage of these terms is specific to research interest or the model or system in use, so they are defined for this study as:

- Passively reversible fouling is permeability loss that does not require any mechanical or chemical intervention on behalf of the operator or controller to reverse. For the sake of a continuously gas sparged AnMBR, the mechanical work of the gas sparging, which also serves other functions, is passive. Changes in pH that occur as part of the filtration process or metabolic process of the microbial communities may also result in passive permeability recovery. A foulant's passive reversibility does not necessarily indicate that it will be reliably reversed passively. It may end up consuming resources targeted at more persistent foulants or require intervention for the sake of timely recovery.
- Mechanically reversible fouling is permeability loss that is reversed by a mechanical interruption, such as a backpulse to dislodge cake foulant.
- Chemically or electrochemically reversible fouling is permeability loss that is reversed by a chemical or mechanical and chemical interruption, such as a backpulse of a cleaning solution resolubilize scalant salts or oxidize organic matter adhered to the membrane surface, known as Chemically enhanced backpulse (CEB).

- Irreversible fouling is permeability loss that is not easily reversed by passive, mechanical, or chemical means such that it would eventually become economically preferable to replace the membranes than to attempt recovery and continued use.

Where there may be a component of any fouling that is reversible by any of the listed means and a component that is not. For this study, where the term fouling is used without further specification, it is in reference to the total effect toward lost permeability of all fouling types and reversibility levels. The methods used to manage and reverse fouling are discussed in greater detail in the following section.

Fouling is typically expressed as a resistance term R (m^{-1}), as a loss in permeability (%) or as a fouling rate term such as the fouling index ($m^{-1}s^{-1}$). It is rarer for fouling to be expressed in terms of energy, power, or pressure. In this study, fouling will be expressed primarily in terms percent loss in permeability, which is useful for daily operation for understanding system performance and membrane health, and power, which is useful for modeling, understanding fouling management principles, and energy optimization efforts.

(1) Modified Fouling Index[42]:

$$MFI = \frac{\mu \alpha C}{2TMP * A^2}$$

(2) Fouling Resistance Model[42]:

$$J = \frac{TMP}{\mu(R_0+R_I+R_C)}$$

(3) Classical Filtration Model[43]:

$$\frac{d^2t}{dV^2} = k(\frac{dt}{dV})^N \text{ or } \frac{dR}{dV} = kR^N \rightarrow R = [kV(1-n)+c]^{\frac{1}{1-n}}$$

(4) Duclos-Orsello Comprehensive Fouling Model[42], [44]:

$$\frac{Q}{Q_0} = (1 + \beta Q_0 C_b t)^{-2} * \exp\left(-\frac{\alpha C_b J_0 t}{1 + \beta Q_0 C_b t}\right)$$

$$+ \int_0^t \frac{\frac{\alpha c_b J_0}{\left(1 + \beta Q_0 C_b t_p\right)^2 * \exp(-\frac{\alpha C_b J_0 t_p}{1 + \beta Q_0 C_b t_p})}}{\sqrt{\left[\left(\frac{R_{p0}}{R_m}\right) + (1 + \beta Q_0 C_b t)^2\right]^2 + 2(\frac{f' R' TMP'^{C_p}}{\mu {R_m}^2})(t - t_{p)}}} dt_p$$

Where equation (1) is a standard in industry and is adapted from soil limnology using microfiltration membranes, (2) is a useful and intuitive conceptual model that is easily applied to get functional fouling resistance term as R_I+R_C, though separation of the two terms is vital as R_I is typically less reversible than R_C (3) generally describes fouling behavior based on blocking modes with loss of fidelity to actual operation parameters, (4) seeks to describe mechanistically the resistance components but in so doing creates an unwieldly equation that, while valid for small microfiltration (>0.2 µm) under constant pressure where specific resistance (*f'*) is known and foulants can be reasonably represented as spheres or as a uniform cake, may fail to adequately describe other scenarios.

1.D. Fouling Control in AnMBR Systems

Current fouling control strategies include mechanical, chemical, electric, and some combination thereof. Backpulse, shearing by fluidized granular activated carbon (GAC) and gas sparging fall primarily into the mechanical cleaning category. Traditionally, gas sparging is assumed to provide cleaning benefits by shearing cake foulants from the membrane surface. However, some studies have demonstrated that shear alone does not explain the cleaning capabilities, and that some of the energy required to remove foulants is imparted by pressure differentials created by motion of the bubbles causing suction [45], [46].

Backpulse with pure water serves to both mechanically dislodge fouling layer while also restoring the repulsive charge of the membranes. Chemically enhanced backpulse (CEB) combines the mechanical cleaning with oxidative, complexing, or acidifying agents. These methods, cyclic backpulse, CEB, and gas sparging are popular in industry for their simplicity and effectiveness and are employed in this study, as findings on their optimization are most likely to be useful in practice.

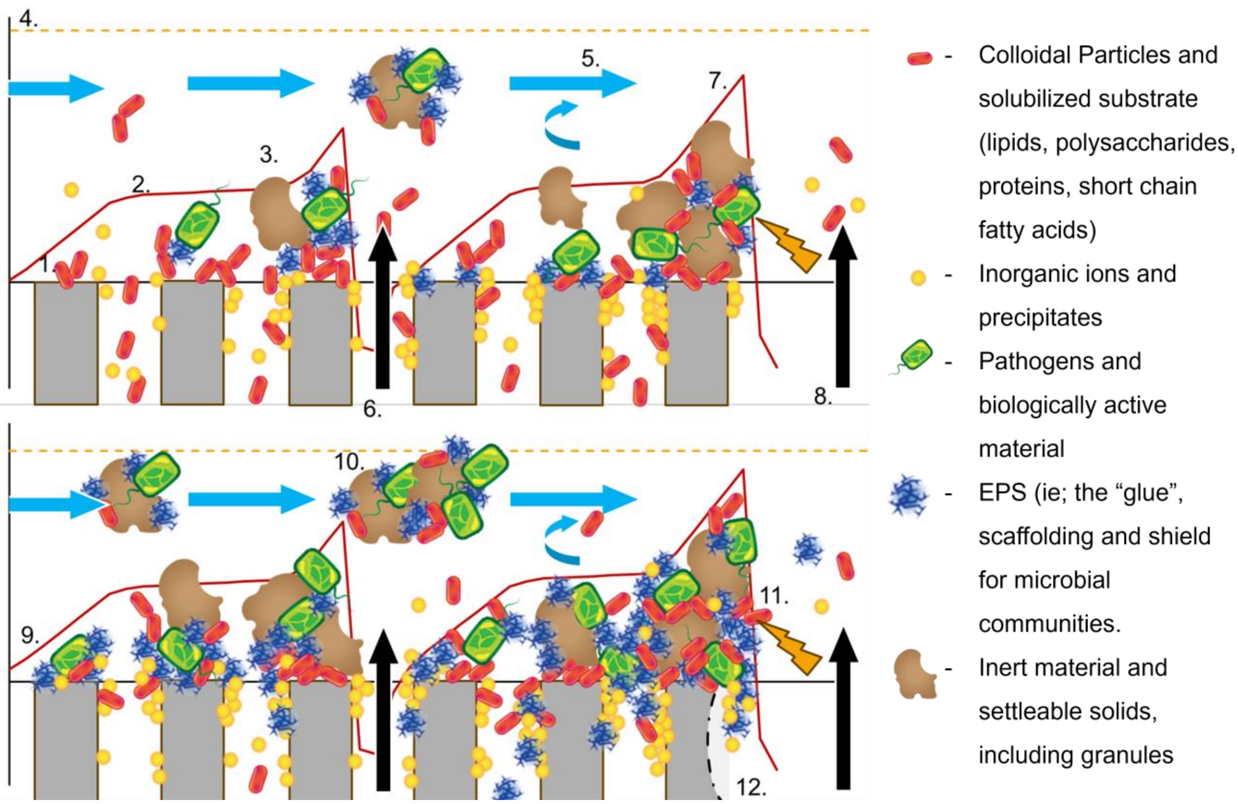

Figure 2. Conceptual Fouling Diagram on anaerobic membrane surfaces

1. Initial TMP Rise at constant flux indicates that strong form critical flux is exceeded, and fouling will occur progressively. Repulsive charges and dispersive forces are overcome by driving pressure.

2. TMP levels off to begin a gradual, generally linear increase as resistance from foulants increases in proportion to colloidal solids rejection.
3. Terminal TMP spike begins as thick layer of accumulated colloidal solids are compressed into a low-water, low-porosity gel and larger particles begin to agglomerate, causing total pore blockage.
4. Operational Max TMP, ie; TMP at which membrane damage is probable multiplied by some design factor < 1.
5. Fouling management force, such as some combination of shear, turbulence and suction from fluidized GAC, high-rate cross flow, gas sparging, or vibration that inhibits deposition of particles, imparts energy to dislodge particles, and transports particle away from the membrane surface.
6. Backpulse to mechanically dislodge cake foulant, which may begin to include granules and other larger particles consisting of inert material, organic substrate, and microbial communities. These mechanical management methods do little to reverse fouling caused by inorganic pore constriction, and, in the case of backpulse with permeate, may increase deposition.
7. With each forward pulse and backpulse cycle, residual fouling increases initial resistance and propensity for particles to agglomerate and shortens the time till major blockages and terminal TMP spike begins, resulting in greater maximum TMPs.
8. Eventually, high TMP or end of a run may trigger a chemical maintenance clean or other fouling management interruption of normal operation. Bleach and Citric acid are commonly used, though other acids, oxidizers, chelates, pure water and other compounds

are also used. May instead be an induced electric field to repel negatively charged particles from the membrane surface.

9. Over time, chemical cleans and other maintenance interruptions are unable to reverse a cumulative portion of fouling resistance and startup TMPs increase with decreasing permeability. Note that biofilm and scalants comprise the majority of this irreversible fouling and may reinforce each other.
10. Solids build up in reactor, providing increased interfaces for microbial communities to metabolize substrate and increasing fouling propensity, though large granules may prove easier to dislodge. Noticeable recovery can occur from such sloughing events.
11. TMP nears or reaches the cutoff level and typical maintenance cleans are likely no longer sufficient due to the structural nature of advanced fouling insulating individual components.
12. A more disruptive, more resource intensive recovery clean, such as a chemical soak may be required, using higher concentrations and long contact times. Over time, processes such as these can damage certain membranes, decreasing their effectiveness as filtration devices, for instance, depolymerization in PVDF membranes.

Part of the goal of a fouling management system is to optimally time cleaning interruptions such that time, energy, and resources expended have the greatest impact to reduce fouling and prevent situations hazardous to further operation. It has been noted that sudden spikes in fouling can correspond with high colloidal loading from the influent and content within reactor, which aligns with general understandings of critical flux and may indicate knock on fouling effects[20], [47], [48], [49]. In particular several studies have revealed that humic acids (HA) have a propensity to cause irreversible fouling, particularly where they can form complexes with Ca^{2+} and other metals [48], [50], [51]. HA are naturally occurring substances in water and are complex macromolecules generated by the incomplete degradation of organic matter of plants and animals and the repolymerization of simple organic compounds such as amino acids, fatty acids, polysaccharides, phenols and other aromatic carbons. They are associated with anoxic environments in soils and are known to enhance binding of colloidal substances and act as catalysts and nutrient sources for microbial communities. It has been demonstrated that oxidants such as bleach can be effective at reducing the fouling potential from HA[51]. Aside from heuristic models proposed for the prediction of fouling, monitoring of known fouling enhancers such as HA may be a path toward fouling prediction and cleaning optimization [20], [42], [49], [51]. The effects of HA and high colloidal loading have not been widely characterized for AnMBRs treating swine wastewater.

1.E. Existing Systems & Challenges

Covered lagoons (approximately 872,000 swine) and complete mix systems (approximately 117,000 swine) still comprise the majority of anaerobic digestion systems for managing swine wastewater in the US [52]. Challenges with covered lagoons include large footprints, difficulty in monitoring and process control posed by variable conditions across the lagoon area, methane slip in the form of dissolved methane in effluent and gas leaks, long HRTs, and poor settleability of biomass leading to SRTs tied to HRT and thus inconsistent or insufficient pollutant removals, and vulnerabilities to weather events such as those experienced in North Carolina. Complete mix systems on the other hand can achieve better removals and more consistent performance, but still do not provide treatment to the level of water reuse while often requiring energy investment to maintain reactor temperature in an optimal range of approximately 35 ^{o}C.

AnMBR treatment of swine manures is still largely in the experimental stage and poses challenges of its own. AnMBR systems, as well as being defined by their operating size, organic loading rate, flux, HRT, SRT and temperature range, are mostly distinguished by their membrane material, membrane geometry, membrane pore size, overall configuration, and fouling management techniques. In continuously gas sparged (CGS) AnMBRs, gas sparging accounts for 50-70% of the energy input to the system, and it is assumed to have minimal effect on reversing or managing pore constriction. Fluidized GAC likely achieves greater shearing forces and can supply interfaces for microbial communities to achieve superior treatment at lower HRTs, but has been shown to result in decreased ability to achieve high fluxes at high volatile solids concentrations, and possible damage to membranes[53].

Two of the most commonly used chemicals for CEB are bleach and citric acid[54], [55], [56], [57], [58]. Bleach, however, is known to create harmful byproducts when oxidizing natural organic matter, known in wastewater treatment as disinfection byproducts (DBPs), namely HAA5s and THMs, which may pose cancer and reproductive health risks as water pollutants. Both citric acid and bleach can represent significant cost and environmental impact to produce[59].

Membrane replacement is another large cost representing a drawback to AnMBRs. High concentrations of acids, bases and other chemicals can damage some membranes. Prolonging the life of the membranes while minimizing the chemical footprint and moving away from those that produce harmful biproducts will be an important part of producing viable AnMBR systems.

There are three typical configurations for AnMBRs, those being; external flow MBRS using membranes that are not submerged in mixed liquor; sideflow or partially submerged MBRs which carry out the majority of the biological activity in a digester, typically a CSTR separate from the membrane chamber, but circulates the reactor sludge through the membrane chamber to produce permeate, and completely submerged MBRs where the membranes are submerged in the reactor sludge in the same chamber where the primary anaerobic digestion is being carried out. External flow (EF) systems usually use tubular or flat sheet membranes that can pull permeate without being fully submerged. Hollow fiber membranes are typically either completely submerged (CS) or sideflow (SS) submerged. High shear cross flow is an easy fouling management strategy to pair with SS, as high circulation rates are typically already involved in SS. In general, SS has advantages over either of the other types as it is more easily modified and allows the operator to isolate the membranes from the biologic process for cleaning activities or other modifications. CS systems, on the other hand, have the advantage of being simpler to build and operate with smaller footprints.

Table 2. Sample of Existing Systems for Comparison

System Type	Config.	Membrane Type	Scale	Waste Stream	Removal -(%) /inf COD (mg/l)	HRT /SRT (day)	Cleaning	Ref.
AD	Covered Lagoon	NA	Full	Flushed Dairy	59.7 /4,900	>25 /NR	NA	[60]
AD	2-Stage Upflow	NA	Pilot 2900L	Swine Food waste-Mix	73-74 /10,000-30,000	11-16 /=	NA	[61]
AnMBR	SS	HF-UF PVDF	Pilot 1720L	Municipal Wastewater	72-97 /207-455	0.11-0.15 /6.2	FGAC 5:30 Relax	[62]
AnMBR	SS	HF-UF NR	Bench 16.5L	Swine Dilute	96 /13,476	13 /45	Semi-CGS	[63]

AnMBR	EF	T-UF/NF PAES	Bench 6L	Swine Direct	96 /1,000-3,000	6 /NR	HSXF	[64]
AnMBR	SS	HF-UF PVDF	Lab 30L	Swine Direct	96 /7000-8000	NR /NR	R-Handwash R-CS$_{(NaOCl500)}$ R-Sonic	[65]
AnMBR	Upflow CS	FS-MF PTE	Bench 3L	Swine Direct	80-95 /15,000-35,000	10-20 /60	CGS 2:8 Relax M-Backpulse	[66]
AeMBR	SS	FS-UF PAN	Lab 28L	Swine Dilute Mix	99.6 /34,200	3-6 /NR	Aeration 1:9 Relax	[67]

HSXF: High Shear Cross Flow
CS: Chemical Soak
CGS: Continuous Gas Sparging
FGAC: Fluidized Granular Activated Carbon
R- : Recovery Clean
M- : Maintenance Clean

1.F. Study Objectives

The goals of this study are a). to characterize the effects of membrane fouling during AnMBR operation with real swine wastewater and evaluate the impact of membrane chemical cleaning frequencies on membrane permeability b). to demonstrate using real swine wastewater the feasibility of swine wastewater treatment in an AnMBR at 20 ^{o}C with efficient carbon recovery as methane, and c). to identify opportunities to optimize the operation, such as robust modeling, that will be transferable to other systems while producing high quality permeate for downstream nutrient recovery and subsequent reuse. These goals were addressed in two separate phases. Phase I focused on variation of cleaning frequency, goal a, while Phase II focused on goals b and c. A physical lab-scale, gas-sparged AnMBR was used to collect the data.

The System operation was also a necessary component to attached research goals as a part of a larger team as permeate from the System was used in other studies as part of a US Department of Energy resource recovery project (DE-EE00009504).

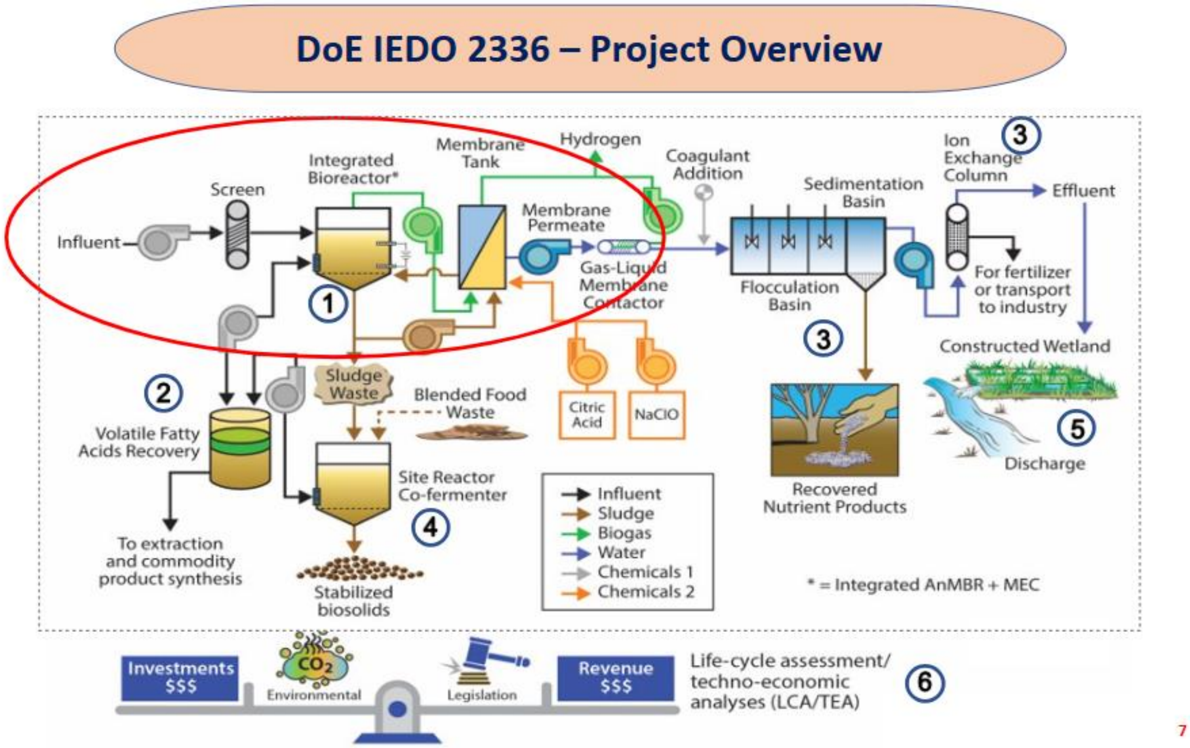

Figure 3. DE-EE00009504 (part of the DoE IEDO 2336 program) graphical overview. Circled in red is the component of the project addressed in this thesis.

Thus, while pursuing the goals of this study with highly variable swine wastewater influent, it was also occasionally necessary to prioritize permeate production, thus overall system performance, over the goals of this study. The impacts of this necessity will be discussed in more detail in following sections; however, it is a credit to the System itself that it functioned this way as part of a larger process train with real goals and restrictions as would exist on a pilot scale or active wastewater treatment facility.

Chapter 2 - Materials & Methods

A lab scale AnMBR (the System) was operated for two periods of 244 and 114 days indoors in a controlled environment. The reactor consisted of a 114.7L digestion tank, a submerged ZeeWeed 2.5 membrane module (nominal pore diameter 0.04 microns, 0.23 m^2, Suez, ON, Canada), a GJ 120 series reversible magnetic gear process pump (MicroPump, WA, USA) used forward draw and backpulse of permeate, a Laboport UN 816 double diaphragm air operated pump (KNF, NJ, USA) for sparging of membranes with biogas pulled from the headspace, a Masterflex iDrive peristaltic pump (Avantor VWR) for feeding of influent swine waste, and a PLC and FactoryTalk control constructed by Rocco Mazzeferro Inc. (ON, Canada) and desktop for interfacing with the system.

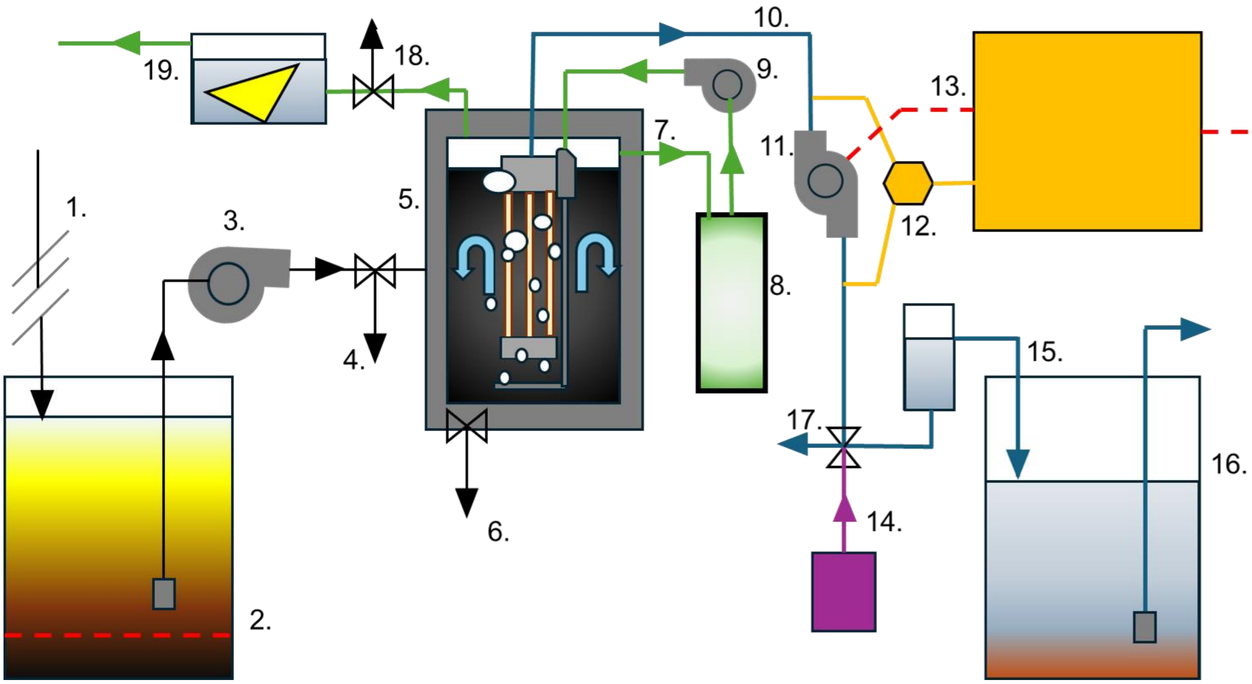

Figure 4. Lab Scale AnMBR Schematic

1. Influent is collected from KSU Animal feeding facilities, screened through #16 mesh.
2. Influent line above confined settling layer of feed stored at room temperature.

3. Peristaltic feed pump.
4. Feed sampling port. Mixed liquor sampling and wasting port.
5. 114.7 L reactor chamber with submerged membranes and biologically active sludge under anaerobic conditions.
6. Settled sludge sampling and wasting port.
7. Headspace Connector and overflow.
8. 20L biogas headspace and overflow chamber. Additionally reduces vapor in the sparge pump intake.
9. Air operated double diaphragm pump delivering 12-14 LPM biogas flow to sparge membranes and mix reactor.
10. Permeate Line
11. Reversible production pump provides driving pressure to produce and backpulse permeate as well as backpulse cleaning chemicals.
12. Pressure Sensor for reporting TMP.
13. PLC receives pressure data and communicates between reversible pump and desktop allowing automatic backpulse and relax.
14. Chemical containers for backpulsing bleach and citric acid during maintenance cleans.
15. Permeate column for stabilizing pressure and maintaining anaerobic conditions during backpulse. Gravity feeds to permeate container.
16. Permeate is stored and used for downstream constructed wetlands for water quality and nutrient recovery experiments. Some precipitation of salts occurs.
17. Permeate sampling port for AnMBR performance analysis and downstream experiments requiring fresh permeate.

18. Biogas out and sampling line for GC analysis.
19. Wet tip counter for biogas metering. Water seal maintains anaerobic conditions.

Swine wastewater was collected from a lagoon draining the Kansas State University animal feedlots swine operation during Phase I. During Phase II, samples were instead taken directly from the temporary pit without dilution from precipitation or runoff in order to achieve consistently higher COD and VS loading. During periods of high pH in the influent (pH≥8.0), pH of influent batch was adjusted to an optimal range (7.3≤pH≤7.6) by HCl addition. No NaOH or other buffer was added to either the influent or the reactor to increase alkalinity. During operation, influent was fed coincidentally with permeate production in. Between each discrete production period (run), the reactor was left in batch. Each run consisted of a series of production cycles which themselves consisted of forward permeation through the membranes to treated effluent receptacles and backpulse of permeate liquid through the membranes to dislodge cake foulant from the membranes followed by a relax period before initiating the cycle again. These steps were different lengths in the different phases as will be described in their respective chapters, but are collectively referred to as a production cycle. The membranes were sparged with biogas from the headspace continuously at approximately 12-14 l/min, including during batch periods. Throughout the course of the operation, feed influent was characterized, and reactor performance assessed according to the following parameters: Chemical Oxygen Demand (COD) removal, day 5 Biological Oxygen Demand (BOD5) removal, Total Solids (TS) and Volatile Solids (VS), Gas Volume, Gas Composition, Hydraulic Retention Time (HRT), and Trans-membrane Pressure (TMP). Turbidity and coliform testing were performed intermittently during Phase I, and more consistently during Phase II. Due to material limitations, sulfate testing of the influent and permeate were limited to only a few samples early in Phase II.

2.A. COD.

The influent and effluent chemical oxygen demand were measured using Hach Method 8000, which is based on Standard Methods 5220 D. Samples for COD analysis were taken weekly. Typically, permeate samples were taken from a glass carboy that was drip-fed from the end of the effluent line. Typically, influent samples were taken from the end of the influent feed line to capture the actual influent quality as it entered the reactor. Influent and effluent samples were collected on the same day after at least an hour of operation and compared directly to determine COD removal. Soluble COD (sCOD) was measured according to Hach Method 8000 after filtering diluted samples through 0.45-micron fiberglass filters. All dilutions were performed using double de-ionized water. In Phase I, small influent samples of 20-40 liters were acquired and COD was analyzed once and assumed to remain constant throughout its use as samples were not retained for more than 48 hours. There was less variation in COD sampled from the lagoon, and so during some periods, COD was assumed to remain constant. In Phase II, Influent was obtained once weekly and used for all runs within a seven-day period. To account for the degradation of COD and VS in the influent over this time, COD was analyzed on the day it was acquired and after five days, and estimated for each day based on the formula,

$$COD_d = COD_0 * e^{-kd}$$

Where COD_d is the COD on the day of the run

COD_0 is the initial COD on the day the influent sample was acquired

$-k$ is the estimated decay constant

d is the number of days since the influent was acquired on the day of the run

2.B. BOD5.

The 5-day, Biochemical Oxygen Demand (BOD5) for influent and effluent were measured according to a modified version of standard method 5210B, in which no seed addition was made given the high strength nature of both influent and effluent compared to municipal water[20]. An iBODpro Dissolved Oxygen (DO) meter (YSI Instruments, USA) was used to measure the initial and final DO for each BOD sample bottle.

2.C. Solids.

Samples for solids analysis were sampled in the same manner with COD in the case of influent and effluent. Sludge was sampled from a port at approximately mid-height of the reactor. 50-ml of sludge were wasted to obtain each sample. Solids samples for TS and VS were analyzed according to a modified version of Standard Method 1684, in which 5-ml of sample were aliquoted into ashed tins and dried at the standard temperatures in triplicates.

2.D. Trans Membrane Pressure (TMP).

TMP was measured continuously during operation using a Endress+Hauser pressure sensor (Reinach, Switzerland) with transistors placed across the suction and pressure sides of the permeate process pump and reported to a desktop computer file in 5-second increments using FactoryTalk View SE (version 10, Rockwell Automation, WI, USA). Raw TMP data was processed into average and largest-magnitude positive and negative values for each discrete period of production (run). Negative values represent forward permeation while positive values represent backpulse.

2.E. Flux

Net average flux (flux) was derived from the measured volumes of permeate produced and the recorded start and stop times of production. Flux was determined for each run.

$$\bar{J} = \frac{V_{Run}}{T_{Run} \cdot M}$$

Where: J is the net, average flux in l/m^{2}hr (LMH)

V_{Run} is the total volume of permeate liquid collected during a run in liters.

T_{Run} is the period of time from beginning of run to end of run in hours.

M is the nominal membrane surface area in square meters.

2.F. Permeability.

Net average permeability (permeability) was derived from the calculated average flux and the averaged production TMP for each run.

$$\phi = \frac{\bar{J}}{p}$$

Where: ϕ is permeability in LMH/bar, and

p is the absolute value of average production TMP in bar.

The initial permeability of the system was estimated based on the average result of 4 clean water flux step test using tap water on virgin membranes. Flux was increased at intervals of 2.174 LMH from 15.217 LMH and then decreased in standard steps to 14.130 LMH. The maximum flux setting for the net flux step tests was 33.700 LMH as it was not anticipated that any flux above this would be used in real operation. After each net flux step test, which ran for two cycles, a high flux test was conducted according to manufacturer guidance in which TMP/Flux values were iteratively predicted and tested to determine a) the flux at which TMP would reach 35 kPa within three minutes and b) a flux at which TMP would no longer stabilize within three minutes. An initial permeability was determined both for clean water and wastewater.

2.G. Fouling

In this study, fouling is examined as a percentage loss of initial permeability in wastewater, and as power spent overcoming fouling which is derived from the difference in expected Q at a given TMP assuming permeability is equal to the initial permeability, versus the observed Q at the given TMP. Thus:

$$F_{\%} = \left(1 - \frac{\phi}{\phi_0}\right) * 100\%$$

and

$$F_P = -(Q_{net} - \phi_0 * M * TMP) * TMP$$

2.H. HRT.

Hydraulic retention time is most often calculated as the volume of a system divided by the flow rate through that system averaged over a period of time. In the case of this operation, a finer calculation method was required due to the dynamic nature of the flux (and thus flow rate) and the inconstant nature of production. While preliminary reports on the data gathered during this period used V_T/Q as HRT, here HRT is calculated using a weighting method as:

$$HRT_i = \frac{1}{2} \cdot \frac{V_{Run,\,i}}{V_{T,\,i}} \left(\frac{T_{Run,i}}{24hr}\right) + \left[\left(1 - \frac{V_{Run,\,i}}{V_{T,\,i}}\right) \cdot \left(HRT_{i-1} + \frac{\frac{1}{2} T_{Run,\,i} + T_{Batch,\,i}}{24}\right)\right]$$

This is a partial series expansion where the computation is enabled by the ability to enter, maintain, and reference each entry in the series in Microsoft Excel on a run-by-run basis. Using the computer hardware and software available, it is possible this way to capture a truer picture of the HRT than the simplified V/Q estimations that are often used for semi-continuous reactors when such estimations are only representative for fully continuous reactors. This calculation assumes that the reactor is completely mixed and that the total volume of influent is added as a sum of discrete packages. As HRT approaches one day or less or as flow approaches continuous flow (HRT < T_{run}), these are no longer valid assumptions and V/Q becomes the more accurate estimate.

2.I. Methane Production

Biogas production was measured continuously from the end of a line connected to the gas headspace of the reactor using a Ritter tip meter, and the data reported to a desktop and recorded using Rigamo software. To quantify the methane production, gas samples were taken intermittently from the headspace and analyzed with a gas chromatographer (Shimadzu GC-2010). The overall gas volume production recorded following a gas sample analysis was multiplied by the percentage methane obtained from the preceding gas sample analysis to directly obtain the volume of methane produced during that period. Where gas volumes were recorded without a corresponding GC sample, a methane percentage was interpolated. In Phase II, gaps were left intentionally over periods of more than a day so that an interpolated point would be used instead of a measured one. The reactor temperature was maintained at 20^oC +/- 2^oC and so no standardization was performed.

Chapter 3 - Phase I Cleaning Frequency Study

Prior observations demonstrated that membrane manufacturer cleaning protocols may be overly prescriptive. Based on a survey of available membrane operation manuals and values reported in other studies, it was found that cleaning demands for membrane systems tend to fall between an estimated 0.25 L solution /m^2 membrane area and 15 L solution/m^2 with concentrations of bleach (NaClO) between 100 ppm and 1000 ppm and acetate concentrations between 200 ppm – 2000 ppm. Typical maintenance cleaning frequencies are between once a week and once daily for durations of 5 minutes to 5 hours [68], [69], [70].

The system using chemical cleans as part of the procedure that was found to operate with the lowest recommended cleaning demand did so by using an integrated reactor with a small volume relative to the membrane surface area and by regularly wasting retentate, which is not suitable for achieving biologic, and particularly anaerobic treatment. A survey of pilot studies by Shin and Bae notes that some systems entirely forgo regular CEB or chemical soaking. However, their study focused on the effects of gas sparging only and was not intended to compare cleaning chemical cleaning procedures.

To determine what effects decreased cleaning schedule would have in an AnMBR treating swine manure, the System was operated under four distinct periods defined by varied cleaning frequency. All cleans consisted of a 1-minute backpulse of cleaning solution followed by 1 minute of relax repeated five times for 10-minutes total per clean per chemical. The concentrations of cleaning solution used were 400 ppm NaClO, and 800 ppm citric acid. This CIP process, also known as chemically enhanced backpulse, completely removes the chemical soaking step suggested by most manufacturers that requires the majority of the chemical input.

The four cleaning frequencies chosen were one clean per week, one clean per two weeks, two cleans per three weeks, and two cleans per week. In practice, due to the needs to manage fouling and produce permeate for downstream use by other research groups, work around limited scheduling time, and collect real swine manure, these became Case 1, average 1.1x/ week, Case 2, 3x/2 months, Case 3, average 3x/2 weeks, and case 4, average 5x/2 weeks respectively. The question for which an answer was sought was, will cleaning efficiency and net flux respond if cleaning frequency at a reduced cleaning volume of 0.25-L is reduced bellow twice weekly? The presumption is that doing so will decrease cleaning efficiency and net flux and lead to a progressive increase in fouling and that increasing the frequency would improve recovery and average net flux. This was not observed.

3.A. Phase I Methods

For Phase I, influent was collected from the KSU swine lot lagoon. Prior to day 187, wastewater was collected from a wet well in the lagoon using a screened pump when possible, and occasionally collected directly from the surface of the lagoon when site conditions did not permit pumping. After day 187, all influent was collected from the surface of the lagoon near the deep pit outfall. Prior to day 157, a fine mesh filter prescreened influent at the feed line inlet. Afterward, due to higher solids and need for high flow rate, this was removed, and influent was prescreened by hand using sieves down to #20. System startup began February 07 with a 1:1 inocculum of swine lagoon waste and AD sludge from Salina, KS WWTP. Regular operation with data acquisition began April 06 after the reactor was assumed to have stabilized. For Phase I, beginning of regular operation is considered day 0. The system was operated intermittently, for an average of 6.10 hours per run (or 2.23 hours per day averaged for 7 days weekly), for an average permeate production rate of 2.26 l/hr (or 5.28 liters per day averaged for 7 days weekly). During this phase, the forward pulse was 12 minutes, followed by a 2-minute backpulse, followed by a 3-second relax.

3.B. Results and Discussion of Phase I

The results show the system's ability to achieve high removal of COD and BOD with maximum removals of 93.0% and 97.6% respectively, but also highlight the challenges in treating a highly variable and heterogenous waste stream with lower removals than would be expected of AnMBRs treating municipal wastewater and lower removals than have been demonstrated in studies using a controlled high strength influent. In this case, the lowest observed removal occurred during an intentional 100-hr continuous run from day 194 to 199 that resulted in a 5-day spike in the COD loading of 3050% over the preceding average daily load. This corresponded with low solids (8680 mg/l) in the system and a significantly lower HRT than prior operation (3.65 days). Thus, it is believed that a confluence of poor operation conditions led to the drop in treatment performance and that the low COD removal is not indicative of technology limitations. Prior studies have shown that treatment

Table 3. Phase I Influent and Effluent Characteristics

		Influent	# of Samples	Effluent	# of Samples
TCOD	**mg/l**	5208 645 - 49375	40	1143 249 - 4740	40
sCOD/TCOD		0.49	37	- -	
BOD5	**mg/l**	2820 271 – 10580	23	330 35.6 – 2471	22
TS	**mg/l**	3175 1653 – 9940	23	2402 1480 – 4600	24
VS	**mg/l**	1295 447 – 3907	23	716 127 – 2667	23
Phosphorus	**mg/l**	67.7 27.2 - 270	27	60.3 0.7 - 128	28
Ammonia	**mg/l**	453 113 – 1138	15	520 203 - 1008	14

of high strength wastewaters at low HRTs is feasible, and it is therefore likely the suddenness of the spike and lack of microbial mass to metabolize the COD that precluded efficient treatment. As suggested by other studies, the fermentative and methanogenic communities may prove robust and adaptable to a wide range of operational conditions, however, they may require substantial time shift toward different demographics and dynamics, on the scale of days and weeks. Following the drop in performance precipitated by the 100-hr run, the reactor performance began to improve to levels above previous (day 221-244). This is most likely explained by greater solids accumulation and growth of fermentative and methanogenic biomass from metabolizing the large spike in substrate as previous studies generally demonstrate improved treatment efficiency with increasing organic loading rates [66], [71]. Thus, despite an initial drop, the system shows that it can respond and adapt. Typically, studies site an adjustment to substantial changes in organic loading quantity or type of three times the HRT.

Table 4. Phase I Performance

	AnMBR
COD Removal (%)	75.3 55.1 – 93.0
BOD Removal (%)	88.6 59.7 – 97.6
Turbidity (NTU)	39.2 2.1 – 118
Gas Production (l/reactor liter-day)	0.045 0.001 – 0.113
Percent Methane (%)	47.4 29.9 – 62.9
Net TMP (bar)	2.27 0.73 – 4.46
Net Flux (LMH)	10.2 3.5 – 25.4
HRT (days)	19.5 3.7 – 25.1
TS (mg/l)	10734 5093 – 28660
VS (mg/l)	5962 1580 – 19587

Both the turbidity and the solids analysis of the permeate indicate surprisingly high values. It was observed that permeate that was initially clear to the eye upon collection rapidly became cloudy once agitated or exposed to air. It is thus proposed that the reactor liquor may be supersaturated with inorganic solutes, primarily Calcium (Ca), Magnesium (Mg), K, N and P salts that rapidly precipitate as these elements are known to exist in high concentrations in swine wastewater. A change in pH from bulk reactor sludge to permeate could not be identified as the cause of the proposed rapid precipitation due to an inconsistent variation in pH from reactor bulk to filtered permeate. Accumulation of an unidentified salt was noted on the effluent line and containers, particularly during the 100-hr run. It is thus possible that the agitation and the container surfaces themselves provided necessary impetus to overcome thermodynamic barriers to crystallization.

20L of reactor sludge were wasted in two stages following the 100-hr run, and 0.05L were wasted from the vertical-middle port of the reactor weekly for sampling during the phase, resulting in an average SRT of 758 days. The exceptionally high SRT is a result of difficulties encountered in this phase establishing a healthy biomass accumulation, as can be noted from the overall low VS. This is attributed to low COD and TS in the influent as well as low initial inoculum addition.

Similarly, biogas production lagged significantly. Substantial gas production was not observed until day 196, corresponding with the greater COD loading of the 100-hr run. On average, only 6.5% of the COD could be accounted for in the measured methane compared to the 30-75% conversion typically reported in lab and pilot studies. Low biogas generation indicates low biologic activity over all, not just in the methanogenic community, possibly explaining substandard treatment.

The initial wastewater permeability was determined to be 186 LMH/bar. This rapidly declined to below 150 LMH/bar by day 1 of analysis, as was anticipated. The greatest average

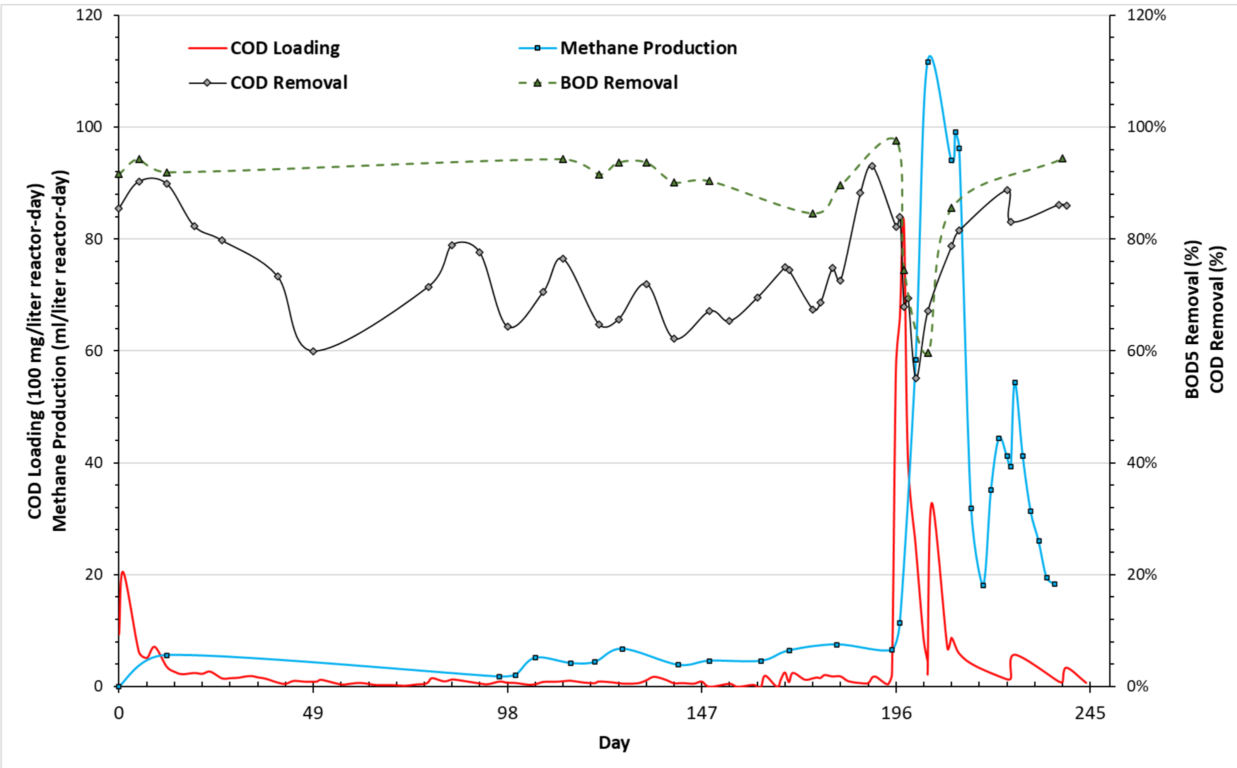

Figure 5. Phase I Process Performance

permeability recovery was observed in case 2 (15%) while the greatest individual recoveries were in cases 2 and 4 (61% and 65% respectively) demonstrating that fouling in the system

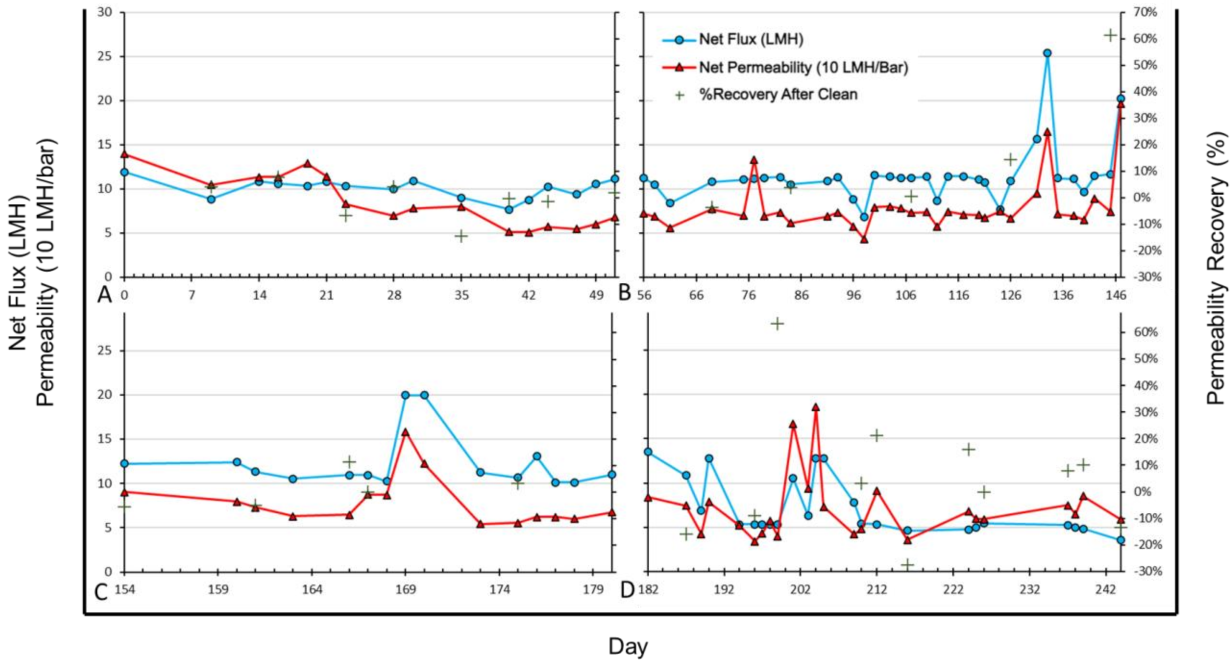

Figure 6. Cleaning Frequency Fouling Response (a) Case 1, (b) Case 2, (c) Case 3, (d) Case 4

continued to be reversible despite long period of operation without maintenance cleaning. For cleaning efficiency, which is the recovered %permeability normalized to the prior %permeability lost, case 2 showed the greatest cleaning efficiency while case 1 shows the least. The results support that chemical cleaning efficiency in a continuously gas sparged AnMBR with backpulse can be retained over long periods without cleans and that cleaning efficiency is not well explained by frequency of cleans alone but will tend to improve with progressive fouling. No strong correlation was demonstrated between cleaning frequency and cleaning recovery. This agrees with previous studies demonstrating that gas sparging is effective at retaining permeability in MF/UF membranes, particularly when it is paired with cyclic backpulsing, and shows promise for application to swine wastewater[54], [58]. A necessary caveat to this finding is that influent COD concentration was lowest during case 2 and 3, and highest during Case 4

No recovery cleans were performed during Phase I. However, fouling Phase I, a recovery clean was performed on the membranes according to manufacturer specifications, with bleach concentration at 1000 ppm, and citric concentration at 20000 ppm, each with a fifteen hour soak in the cleaning solution. A wastewater flux test determined the permeability at 35 kPa to be 127.7 LMH/bar after recovery clean, and an initial run with wastewater found the net average permeability to be 132.5 LMH/bar, showing that the recovery clean is effective in restoring permeability at a recovery of 39.8%. Despite this, permeability was recovered to only 68.7% of the original value, suggesting that 31.3% of the total permeability was lost irrecoverably by the end of operation of Phase I.

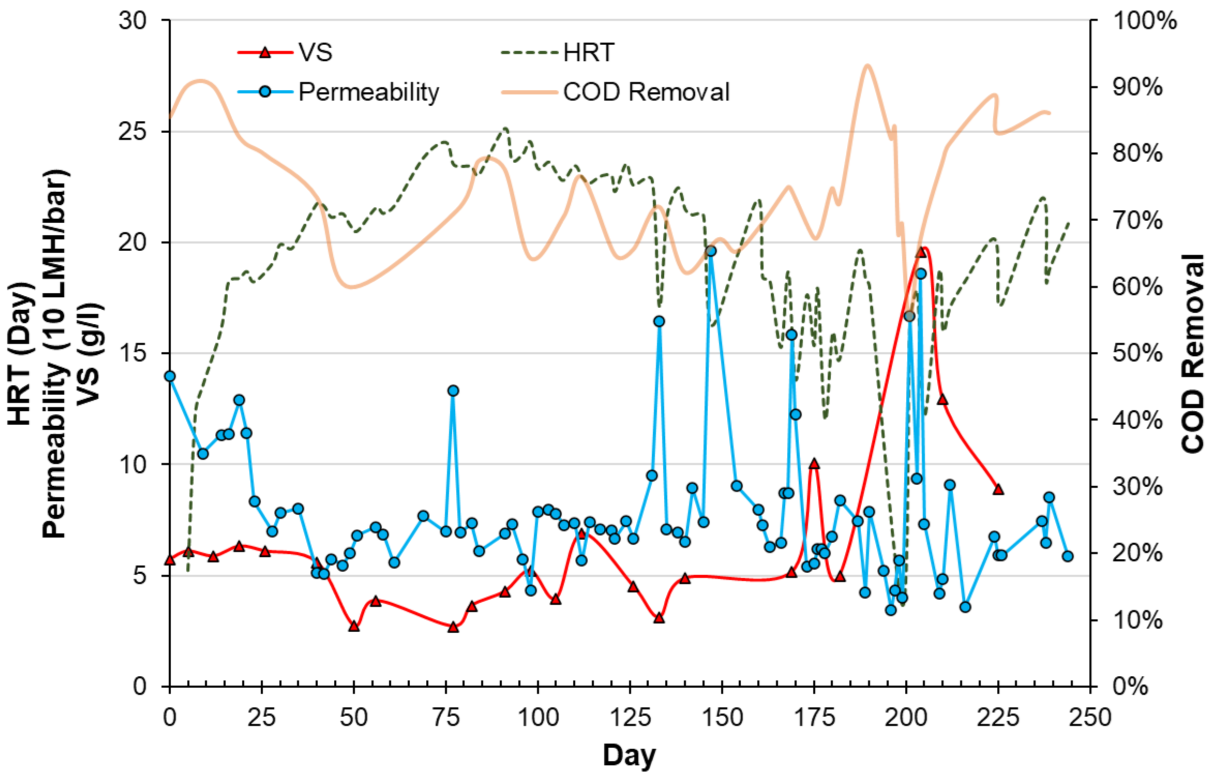

Figure 7. Solids Impact on Process Performance

Chapter 4 - Phase II Verification of Process Performance and Further Optimization

The system was operated consistently for an average of 7.37 hours per run (or 5.29 hours per day averaged for 7 days weekly), for an average permeate production rate of 1.95 l/hr (or 10.3L per day averaged for 7 days weekly). During this phase, the forward pulse was 10 minutes, followed by a 1-minute backpulse, followed by a 1-minute relax. Maintenance cleans were performed twice weekly at a reduced volume of 250 ml per clean. Due to the challenges of phase I, the primary goal for Phase II was to demonstrate treatment and gas production in a reasonable range while assessing further opportunities to optimize fouling management.

Samples for COD analysis were taken twice weekly. BOD5, pH, and VS/TS were taken once weekly. Gas volume and gas composition was recorded each day of the week excluding most weekends; however, data was collected twice on the first and last days of the week to track production over the weekend batch period. Coliform and sulfate removals were assessed only 4 times each due to limited testing supplies.

4.A Phase II Methods

For Phase II, influent was collected from a vat filled directly from the manure storage pit instead of from the lagoon with the goal of consistently treating a higher strength influent than was observed in wet periods with the lagoon. The influent was filtered through a #16 aluminum mesh as pretreatment to remove large solids and grit that could harm the membranes. The reactor was inoculated with 81L of anaerobic digestate from Salina, KS municipal wastewater treatment plant and an additional 27L of swine manure were added with 2L of double de-ionized water to begin startup. On day 11, startup was deemed to be complete when the change in COD of 50-ml samples of permeate decreased to <1% compared to the prior day's. Semi-continuous operation was initiated on day 12 and was terminated on day 114 after the system demonstrated minimal methanogenic activity after a long batch period, necessitating a new inoculation.

4.B. Results and Discussion of Phase II

The results of Phase II demonstrate the system's ability to produce consistent, high quality effluent, with CODs in the range of municipal wastewater. As has been supported by other studies, the comparison of Phase I and Phase II performance demonstrates that AnMBRs are not only capable of handling high strength waste streams, but that the stability of treatment, as well as removal and gas generation efficiency improve with higher average influent VS and TCOD. COD removals and biogas generation lagged from day 11 to day 28. This is attributed to low COD in the startup influent (993 mg/l) and the time required for the microbial community in the reactor to adjust to higher COD loading that would be typical of the operation. From day 28 until operation termination, COD removal averaged 91.9% and did not fall bellow 80%. A drop in COD removal after day 61 follows the second of two solids wasting events on days 54 and 61 in which 4 and 6L of reactor sludge were wasted respectively, showing again that low removal accompanies low solids concentrations. Aside from these two events, 0.1L of sludge were wasted from the reactor each week for analysis, with 0.05L wasted from the vertical middle of the reactor and 0.05L wasted from the bottom where,

Table 5. Phase II Influent and Effluent Characteristics

		Influent	# of Samples	Effluent	# of Samples
TCOD	**mg/l**	9013 837 – 57908	24	372 278 – 553	24
sCOD/TCOD		0.20 0.02 – 0.60	24	- -	
BOD5	**mg/l**	1071 175 – 3197	12	50.2 33.4 – 81.0	12
Phosphorus	**mg/l**	128 80.3 – 237	9	79.6 58.9 – 83.3	7
Ammonia	**mg/l**	420 266 – 764	9	527 369 – 697	10

despite mixing from the continuous gas sparging, solids accumulation and resulting acidification occurred. Solids sampled from the middle port are assumed to be representative of the reactor and reported solids values are from these samples. However, the lower pH observed in the settled sludge from the reactor suggests that fermentation may outpace methanogenesis in the bottom of the reactor (roughly 3 inches depth), as, in a tight syntrophic relationship, methanogens should consume the hydrogen substrate produced by fermenters. Alternatively, acidification in the settled layer may indicate a dominance of methylotrophic groups such as

Table 6 Phase II Process Performance

	AnMBR
COD Removal (%)	88.0 64.5 – 99.2
BOD Removal (%)	90.7 83.0 – 98.1
Turbidity (NTU)	0.44 0.19 – 0.98
CH_4 Production (l/reactor liter-day)	0.104 0.000 – 0.596
Percent Methane (%)	64.1 20.3 – 81.9
Net TMP (bar)	0.227 0.073 – 0.446
Net Flux (LMH)	8.7 6.0 – 17.0
HRT (days)	7.6 4.8 – 26.1
TS (mg/l)	22178 18027 – 25310
VS (mg/l)	14029 10783 – 16500

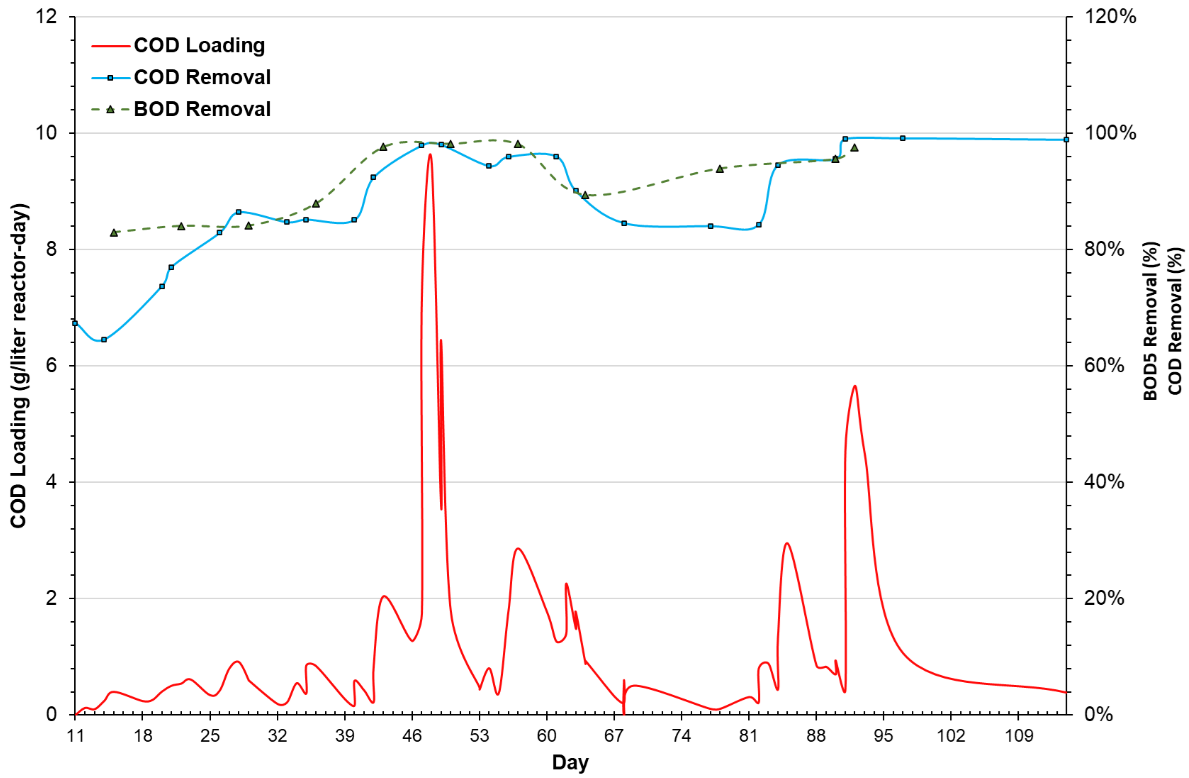

Figure 8. Phase II Process Performance

Methanosarcina. Implementation and control of a recirculation process will likely be a crucial element of this style of reactor should the operator wish to operate the system in fermentative mode to produce long chain fatty acids for extraction and commodification. The average SRT in Phase II was 949 days.

Gas production prior to day 51 is noted to be unreliable due to the metering device maxing out during periods of high gas production. Thus, true gas production is likely higher than reported. A new, wet tip gas meter was installed with a higher limit measuring capacity. The overall, estimated COD conversion to CH_4 is 28.9%. In the period from day 52 to the end of phase I when more reliable measurements were possible, a 45.6% (0.177 L/gCOD) average conversion of COD to methane was achieved. During the most stable period of operation from day 61 to day 93 after the third air exposure and prior to a long batch period, average estimated COD conversion reached 48.4%. Other sources support that conversion from COD to methane can be as high as 50-75% in an AnMBR with methane production between 0.1 and 0.5 L/gCOD removed typically at temperatures of 30-35 °C [70], [72], [73]. Based on the theoretical 0.67 gCOD/gSO4 and a limited number of sulfate analyses, an estimated 8.3% of COD was consumed by sulfate reducing bacteria.

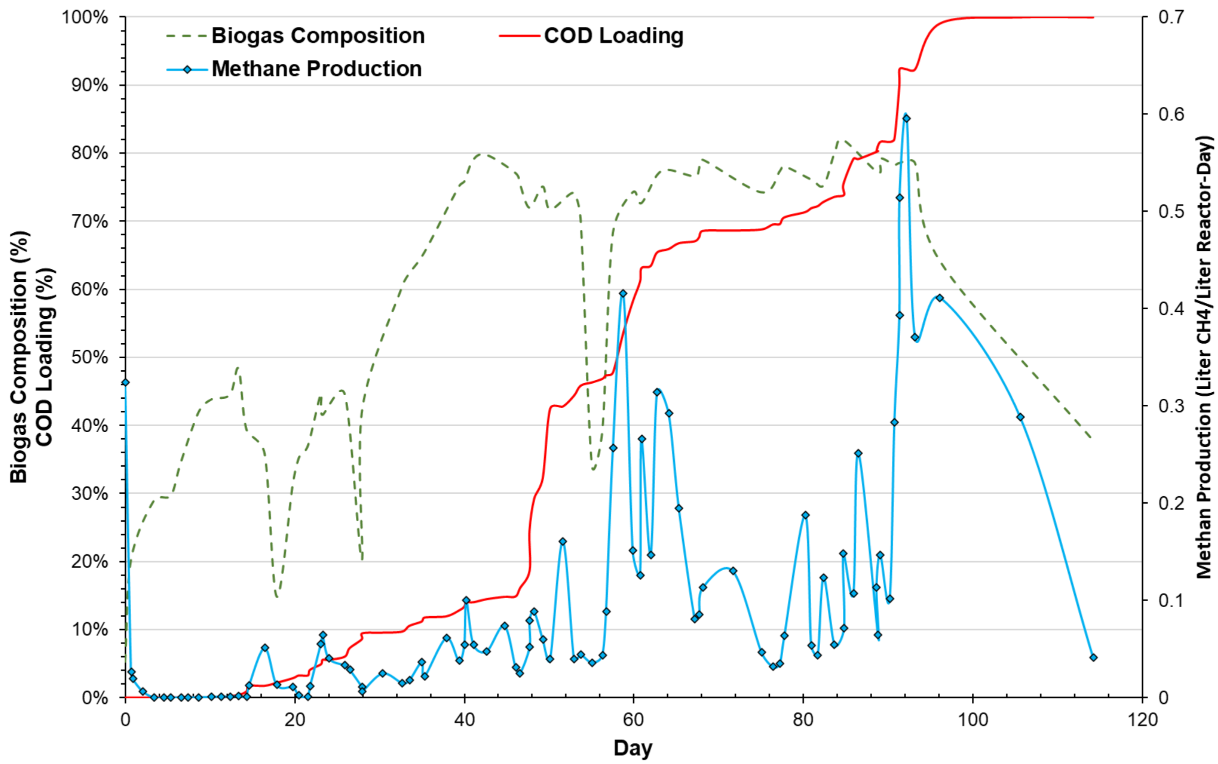

Figure 9. Phase II Methane Production and Biogas Quality with COD Loading

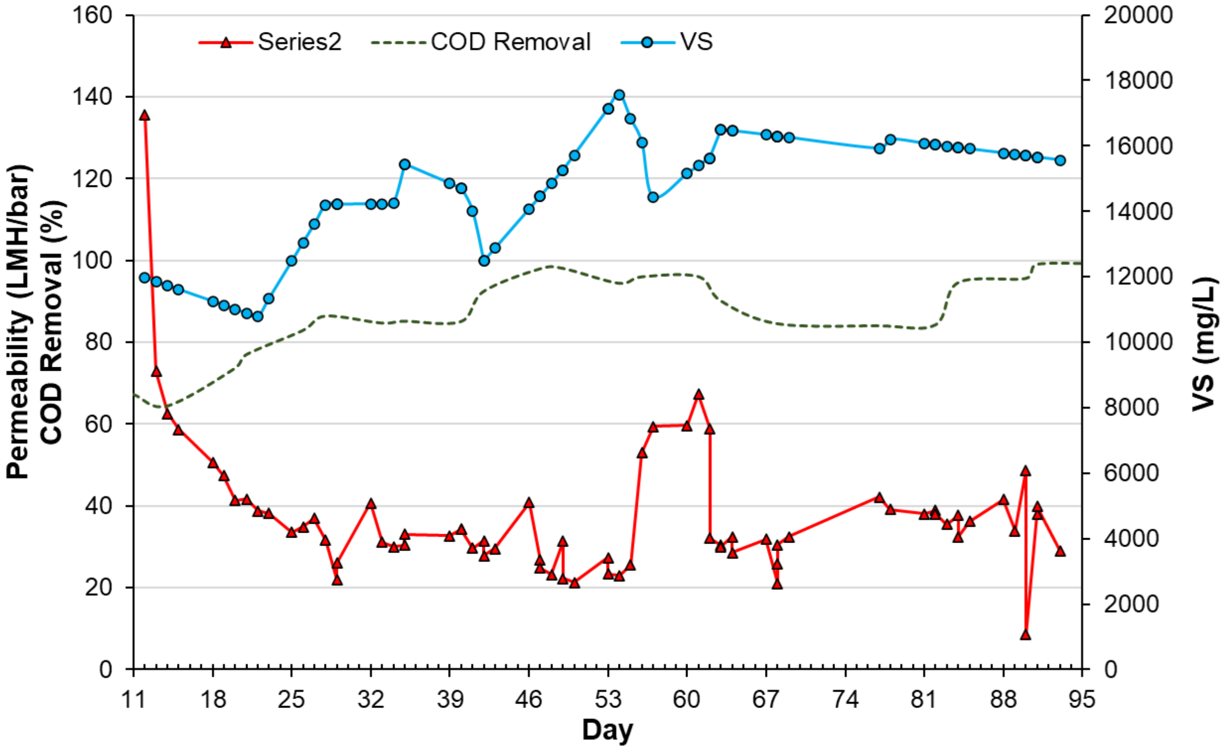

Figure 10. Reactor VS Impact on Process Performance for Phase II

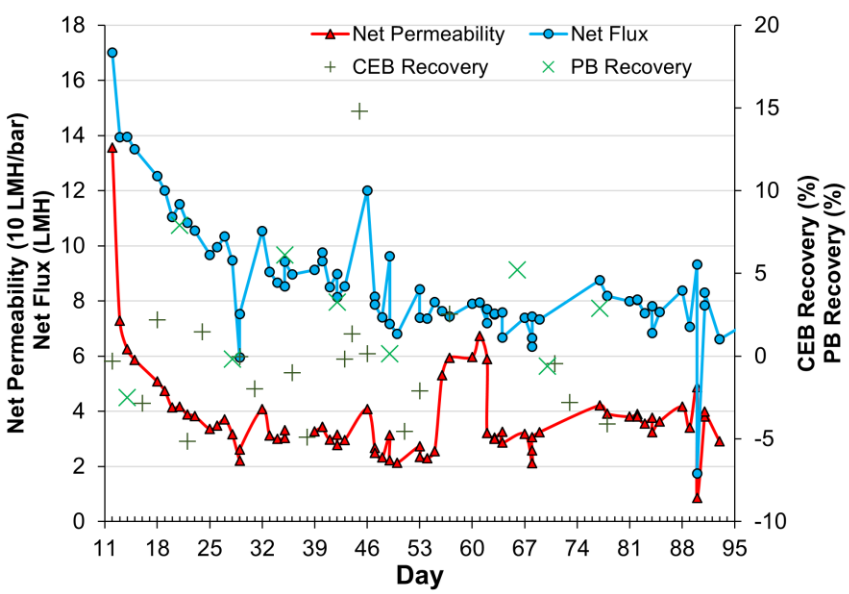

Figure 11. Impact of different cleaning methods, Chemically enhanced backpulse and Passive Batch Recovery on net permeability.

The permeate was not analyzed for dissolved methane fraction, but the high methane percentage in the headspace biogas, 72.6% averaged from day 51-114, supports that sparging of the membranes with biogas enriches the CH_4 content of the biogas by saturating the reactor liquor with the more soluble CO_2. The reactor was exposed to ambient air for 3 different periods following days 16, 25 and 52 and demonstrated good recovery back to anaerobic conditions and CH_4 generation. From day 60-93, during a period of stable operation following the final air exposure event and preceding a long batch mode, the reactor achieved an average biogas composition of 77.4% CH_4, compared to the typical upper limit of 75% for anaerobic digesters. This has implications for energy optimization, as energy spent gas sparging may represent savings in energy required to upgrade biogas to biomethane downstream. During Phase II, substantial permeability recovery was observed following batch periods (weekends). The average chemical cleaning recovery during Phase I was -0.6%, while the average passive recovery from two-day or greater batch periods (PBR) with continuous sparging was 1.8%.

Highlighted by the data from day 56 to 63 is the effect that mechanical operation parameters have on performance efficiency. As fouling began to inhibit performance to the point where dangerous TMP levels triggered automatic shutoff (41.5 kPa), the net flux tests were repeated and a new sustainable flux calculated to be 19.41 LMH at a Q_{set} of 4.464 l/hr. In the interim, the Q_{set} of the system was 3.5 L/hr, well below the previous 5.4 L/hr, possibly approaching a true sub-critical flux. In this transition period, perceived permeability increased drastically without affecting net flux. In other words, energy spent trying to increase flux beyond critical flux is wasted. This is not a surprise as it follows naturally from accepted critical flux theory. But is an important fact for operators and optimizers to be aware of and highlights the need to shift from evaluating permeability at a set TMP to evaluating an average permeability over a range of possible operational Q_{sets} or fluxes. A Wilcoxon test was chosen to analyze the statistical relevance of the difference in observed treatment between CEB and PBR due to the difference in sample size (21 CEB tests compared to 10 PBR tests) and the variance from normal distribution observed in PBR samples under 30-count. The Wilcoxon test is robust to these conditions and supports that the difference between the two observed recoveries is statistically significant within a 90% confidence interval (p=0.069). This supports that gas sparging has a larger impact on maintaining membrane permeability than chemically enhanced backpulse. It also has implications on energy optimization and environmental footprint, as it implies that the toxic HAA5s undoubtedly produced from CIP with bleach may be reduced or eliminated.

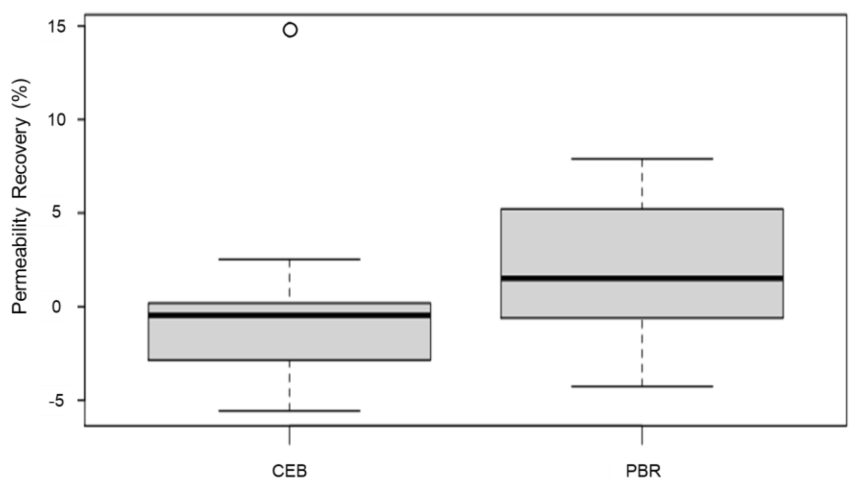

Figure 12. Boxplot Comparison of CEB (n=21) and PBR (n=10)

It is possible to represent fouling in many ways. The way found most immediately useful in terms of daily operation was %permeability lost. However, for the sake of optimization, representing fouling as power spent overcoming it is also useful. Along with the PBR identified during Phase II, it was noted that fluctuations in fouling on a daily basis closely mirrored HRT, which itself reflects the ratio between energy spent drawing permeate and thus

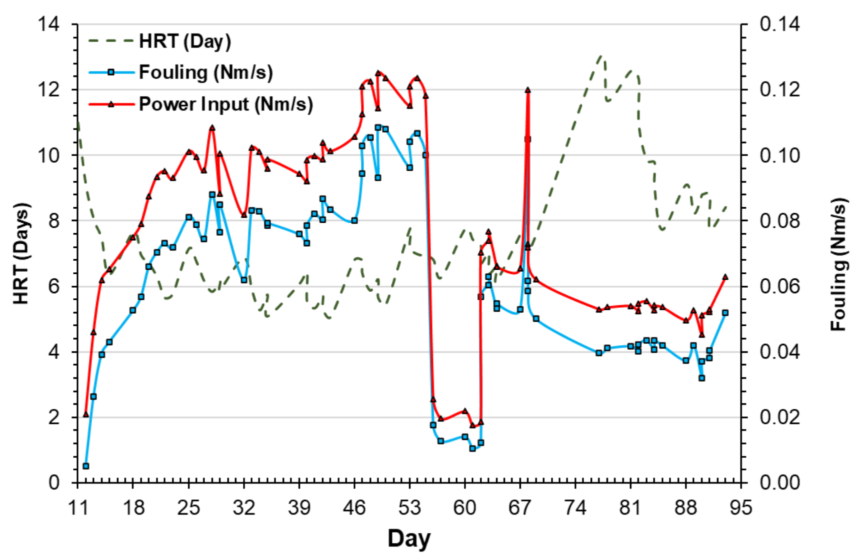

Figure 13. Fouling Behavior with HRT

increasing fouling, and energy spent in recovery during operation and batch. This makes HRT a potentially useful parameter for predicting and controlling fouling behavior in the system. The review of literature revealed only one other study that highlighted HRT as a major factor in membrane fouling and treatment performance, while some proposed AI models appeared to leave out such operational variables altogether [42], [66], [74]. The results of this analysis reveal that, for most target fluxes within the range that could achieve the target net fluxes of 10 LMH or higher, overcoming fouling resistance consumed the majority of power input. This agrees with past studies showing that fouling resistance can account for 80% or more of the total filtration resistance in MBRs [41], [43].

Chapter 5 - Statistical Analysis of Parameters and Model Development

As can be seen from Equation (4) in section 1.C., attempts to describe fouling (in that case reduction in flow over time at constant pressure) using physical and chemical principles can evolve complex formulae that are nevertheless restricted by limitations or require substantial investment into acquiring data to input. Such inputs may ultimately be determined empirically or by reference, lest acquisition entail operations with a physical system that would preclude the need for a model. The models that receive the most attention in practice are those that are simplistic, robust and require minimal investigation to use, yet much of recent academic focus on model development for MBRs has been on those physically based.

Statistical models, on the other hand, have been maligned for their tendency to overfit data without developing any physical basis for their results, resulting in models that are overly specific to the operational parameters from which the test and validation data was derived.

This section will discuss attempts during this study to develop a model that strikes a balance between the two approaches, as there is clearly a need for a model approach that is simple in terms of its inputs and calculations, robust to the range of operational conditions that are encountered by individual systems and operators in the field, and can be readily adopted for multiple systems either on its face or by straightforward, experimental development of parameters. Though the model development was ultimately unsuccessful, the results may yet be useful to future research.

The data from phases I and II were analyzed and used to develop first a conceptual mathematical formula to describe fouling in the system. Six parameters that were varied during both phases were chosen for correlation analysis: CODin (g), solids rejection (N_x, g*m^{-2}), VS/TS ratio, pH, HRT (day), and cleaning frequency (/week), or period (day) in the form:

$$y = a_1 x_1^{b_1} + a_2 x_2^{b_2} + a_3 x_3^{b_3} + \cdots \ a_n x_n^{b_n}$$

This form is of the often maligned, purely statistical kind. However, the goal was to both test the particle swarm optimization scheme developed for the study and determine general fouling responses to the variables in conjunction with simple linear regression with fouling response and individual variables and correlation assessments with pairs of variables

Initial attempts to optimize a formula of this form based on weekly summaries of data from Phase I failed to produce promising results, with %deviations of 90% or greater. Additionally, consistent relationships were not determined as parameters varied widely between runs. Clear challenges with the method used were:

- Flux was an imbedded term in CODin, N_x and HRT
- Fouling was thus implicit in the formula, as increase in fouling would result in lower flux, and higher flux would occur as a result of decrease in fouling.
- Weekly summaries of the data from operation result in loss of fidelity and were prone to error due to gaps in data.
- The regression form used was general and had no first principles or even empirical basis.
- The particle swarm optimization scheme being used is novel, and relatively untested and unrefined, having been created for the purposes of this study.

In conjunction with this initial attempt, Pearson R coefficients were determined for each pair of variables, and it was found that only CODin and N_x were correlated above R=0.5, indicating that one should be dropped from the model. Change in fouling was then plotted against each variable to develop and evaluate linear trends. This further confirmed that cleaning frequency had little predictive power in terms of fouling with $R^2 = 0.004$. CODin produced the linear trend with the lowest R^2, however, it was further assessed based on the knowledge that COD should have a logarithmic relationship to fouling. Total observed fouling in available permeability was plotted against both cumulative CODin and cumulative N_x and a logarithmic trend was applied. It was found that N_x was the better predictor of fouling, $R^2 = 0.2343$ compared to R^2=0.2099. Thus, CODin was excluded as a variable for future models. Cleaning frequency (or period), and pH would also be excluded from the subsequent model as well due to the lack of a clear relationship between the variable and fouling behavior. The subsequent model included separate terms for VS and FS as it follows from the fouling pathways that these would have different fouling impacts. It also included a J_{set} term as varying this operational parameter produced a large effect during Phase II.

For the next model, the simple combination of nonlinear terms was abandoned for a more intuitive form based on observed behavior of the system and the variables that seemed to have the most effect.

$$y = [a_1 \ln(x_1^{b_1}) + a_2 \ln(x_2^{b_2})] * \left(a_3 \frac{x_3^{b_3}}{1 + x_3^{b_3}}\right) * (1 + a_4 x_4^{b_4})$$

Where: y is the power consumed to overcome fouling normalized to membrane area

x_1 is the cumulative volatile solids rejection

x_2 is the cumulative fixed solids rejection

x_3 is J_{set} in LMH

x_4 is the HRT in days

And a_i and b_i d are parameters to allow for the optimization of the formula.

The parameters for this model were optimized using the daily data from Phase II using GRG nonlinear descent in Excel Solver and with the particle swarm optimization scheme in SciLab. The GRG nonlinear optimization consistently outperformed the PSO, even when the PSO was initialized from the GRG solutions, indicating an inherent flaw in the PSOs objective seeking scheme. The lowest percent deviation achieved by PSO on its own was 32.6%. Other studies have achieved good prediction results using PSO and other heuristic optimization algorithms.

Though the formula may seem complex, it is a relatively simplistic attempt to describe fouling in the reactor compared to others. It excludes many physical and chemical processes known to affect fouling behavior with the hope of capturing the most important variables in a robust way. Though many physically derived models have reported very high (>95%) capture rate of real data, the proposed model would be beneficial due to the minimum equipment needed to obtain input variables (just two ovens, a scale and crucible, and a flow measuring device such as a graduated container and digital clock) and the straightforward calculations involved. It has been demonstrated that the overall behavior of fouling takes on a logarithmic, or 90^{o} transformed sigmoidal shape with respect to flux over time, and it was clear from the data that observed fouling impedance was limited by the driving pressure applied, or the flux the system was attempting to achieve. It was expected that the coefficient a_4 would be negative based on observations and solutions for this formulation supported this. Despite promising results, iterations of solver were not initially able to produce a satisfactory model, and so the approach was changed to instead solve

$$y' = e^{y} = [a_1 \ln(x_1^{b_1}) + a_2 \ln(x_2^{b_2})] * \left(a_3 \frac{x_3^{b_3}}{1 + x_3^{b_3}}\right) * (1 + a_4 x_4^{b_4})$$

Where: y is the power consumed to overcome fouling estimated from real data and y’ is LHS term for the objective of GRG.

Such that the final model is written as

$$y = \ln \{[a_1 \ln(x_1^{b_1}) + a_2 \ln(x_2^{b_2})] * \left(a_3 \frac{x_3^{b_3}}{1 + x_3^{b_3}}\right) * (1 + a_4 x_4^{b_4})\}$$

Solver was able to find a solution using this form with a percent deviation of 16.0%. However, the percent deviation bands of this model were only able to describe 61% of the data, and so it was modified by adding a constant, which resulted in a model with a percent deviation of 19.5%. The model prediction +/-19.5% describes only 67% of the data points and so does not meet the requirements set for acceptance of the model. Due to the model’s inadequacy, no validation was performed, though future attempts to create a model for the system of a similar form may find the results of these efforts useful. The model solution model parameters are:

a1 = 7.8754, b1= 16.0041

a2 = 0.2168, b2 = 0.4407

a3 = 2.27E-05, b3 = 1.0171

a4 = 9.3987, b4 = -0.1066

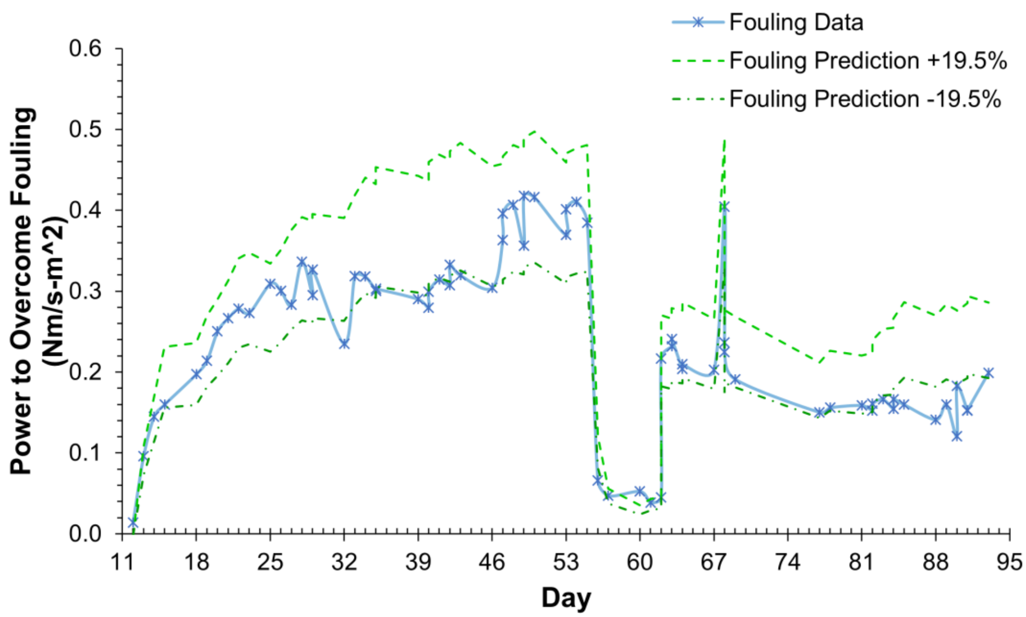

Figure 14. Fouling Prediction Model Performance

In some respects, the model appears to perform well, such as predicting steep drops and increases in power devoted to fouling as a result of varying J_{set}. It can also be seen to follow minor variations in fouling believed to be explained in part by HRT. However, this is to be expected as the formula was developed specifically with the morphology of the data in mind. As a purely empirical model, its inability to predict the values it was trained on to the desired degree makes it invalid. While the intent of the initial J_{set} was that it would be a sustainable flux, the data from day 11 to day 55 reveals that fouling proceeded as expected for a non-sustainable flux, rising sharply and leveling off by day 30, then remaining apparently stable until a sharp rise after day 46. The model fails to predict both the leveling off and the sharp rise, which is expected as this behavior is not observed from the logarithmic function used in the model. A perfect model would exhibit an R^2 of 1 were its real data counterpart plotted against it, as would a model that could be described as a linear transformation of the real data from which a perfect model could be directly acquired. While R^2 is not intended for use with nonlinear equations, looking at Y as a function of prediction y also gives an idea of goodness of fit. The R^2 of the best fit (least squares) line of Y with respect to prediction y is 0.8891, implying numerically what is observed, that while the model fails to predict observed data within the widest reasonable band, it describes the variations or morphology of the data somewhat well.

Early attempts to use hyperbolic sine function in the model failed. A difficulty in trying to model the non-sustainable flux fouling progression with simple functions is that there is, theoretically, a flux below which this behavior is not observed. A super linear behavior such as that exhibited by sinh is difficult to suppress over time. Part of the difficulty with stage 3 fouling is that it is partially dependent on antecedent fouling which could make a more appropriate model implicit with respect to fouling. Either this, or breaking fouling into two separate terms, reversible and irreversible, would begin to work against the efforts of making the model simplistic, though a potential solution to this would be to use the model to project behavior in a series such that antecedent fouling could be referenced. This would likely pose a challenge to optimizing the parameters. Another option that has been explored in other research but was not preferred for this study is piecewise modeling.

Chapter 6 - Challenges, Limitations and Sources of Uncertainty

It is to be expected with wet lab research that things do not always go as planned. If the systems we worked with were perfectly understood by us and offered no surprises, there would be little need to investigate them further. The labscale AnMBR was full of surprises.

Beginning in September the reactor flux began to drop unexpectedly. This was occasionally accompanied by TMP spikes that could be gradual over the course of the day, sudden just within a production cycle or two, or jagged simply showing a lot of instability in a day and making safety of operation uncertain. At this time, there was no automatic cut-off for high TMP and so such behaviors necessitated greater supervision. Possible causes of the erratic TMP behavior and declining flux are:

1. High inorganics in the influent causing greater scaling and clogging with large particles. Through September and October, lagoon was drying up, and more salt crystallization was noted on all reactor appurtenances.
2. Onset of mechanical failures in both the sparging pump and permeate production pump.

Where flagging flux in Phase I was initially attributed to terminal fouling of membranes, it became clear after testing of recovery cleaned membranes and replacement and refurbishment of pumps that much of the fault originated from wear and tear on the pumps. While the AnMBR is often discussed as a biological treatment platform that leverages advanced membrane manufacture technology, it is also a mechanical and hydraulic system where all performance and data will also be affected by the performance of these part. For instance, measured gas production and measured TMP will be affected if the sparging pump creates a positive or negative pressure in the system due to uneven cycling, as occurred in a failed phase.

While replacement of punctured diaphragms in the sparge pump was sufficient to restore its function, the production pump had to be completely replaced after suffering catastrophic failure due to water damage. What caused this for certain is still unknown, though it is possible that crystallization within the pump, as well as aspiration cavitation from dissolved CO_2 and methane caused sufficient damage to allow leakage from the pump head to the solenoid control. It is also possible that a loose gasket allowed moisture to enter the pump drive during regular operation.

Initially, the thesis was intended to focus on sparging control and optimization of the sparging rate and pattern for fouling management, energy use efficiency, and recovered methane maximization. However, limitations in the sparging pump used in the system and ongoing issues with maintaining mechanical performance and acquiring equipment necessary to execute experiments precluded pursuit of these topics.

The data reported in this thesis is provided without uncertainties or errors, largely due to the degree of aggregation in each reported value. For instance, each BOD5 removal shown comes from six, nine or twelve measurements. The uncertainties associated with each measurement are difficult to represent in removal efficiencies or averages due to the fact that removal efficiencies, treatment residuals and influent and effluent BOD5 averages do not follow normal distributions being skewed left. Only the measurement results for individual samples could be said to follow a normal distribution or exhibit random error.

BOD5 determination is an analysis fraught with error due to the heterogeneity of wastewater samples, and the sensitivity and limitations of the testing procedure. It is fair to say that skill of the test conductor plays a role in the success of the test. The average percent deviation for influent BOD5 measurements in phase II was 16.6% with three dilutions excluded from the results because their final DO concentrations were out of range or because their percent deviations were above 33%. The average percent deviation for permeate BOD5 measurements in phase II was 9.6% with only two dilutions excluded due to final DO concentrations being above initial DO minus 2 mg/L. In Phase I, it was not practical to exclude data due to percent deviations as doing so would have limited the number of samples considered to an unreasonably small number. 29 influent dilutions and 30 permeate dilutions were excluded due to being out of range. BOD5 provides a good example of challenges of testing and sampling error, how uncertainties arise and how they were dealt with. Other sources include solids concentration and gas composition data, which were interpolated for points between samples. The dichotomy between Phase I and Phase II data acceptance exemplifies the improvement in analysis and data acquisition in the team over both periods.

In addition to uncertainty from measurements and the propagated uncertainty within derived values, there is, with a complex biophysiochemical system, uncertainty in the relationships between what is observed and cause. In other words, it is often especially difficult to move past correlation to find strictly causal relationships. In particular, Phase I results are shadowed by the ongoing mechanical failures of the system so that it is not known to what extent observed fluxes and thus derived permeabilities were a result of known fouling behavior or degrading pump performance. There are many such uncertainties baked into the data. However, throughout the study, effort was made to quantify and assess error and improve on analysis techniques to limit errors and uncertainties. However, for most aggregate performance data it is believed that a range of values gives the best impression of the variability of the data and that is reasonable for averages to be presented with an adsorbed uncertainty. It is not uncommon to present data for these processes this way.

Chapter 7 - Conclusions and Recommendations for Future Work

This thesis presents data from two phases of operation of a novel scale anaerobic membrane bioreactor treating real, variable swine waste. The total period of operation was 258 days during which approximately 2,349 liters of wastewater were treated. The data supports that, counter to general practice, a healthy microbial system and well operating reactor can sustain OLR shocks due to higher than usual COD and VS concentrations in the influent, and that the reactor can operate to achieve nearly 50% conversion of COD to recoverable CH_4 in biogas at 20^{o} C as opposed to the optimal 35^{o} C, and that it can do so with limited to no other chemical pretreatment of the influent or amendments to the reactor liquor. While it is often emphasized how delicate the survival of methanogenic communities can be, the results of this research show that they can also be tenacious, which should come as no surprise given their preeminence and longevity on this planet.

The study focused on the long-term fouling behavior of the reactor, which used ultrafiltration, hollow fiber PVDF membranes with continuous gas sparging and cyclic backpulse and relax as its primary fouling management strategies. It was found that variation of chemical cleaning frequency did not have a statistically relevant effect on permeability recovery, whereas extended batch periods of two days or more showed substantial recovery. Other studies have shown the continuous gas sparging is an effective fouling management strategy, and this study extends these findings swine AnMBR operation.

By pursuing granularity in representations of the process performance and operational parameters, it was found that small variations in observed fouling resistance closely mirror small variations in HRT and target instantaneous flux on the scale of days. This was leveraged in an attempt to create simple, robust, and easily adaptable model for prediction of fouling behavior that, while its morphology in comparison to that of the real data was promising, ultimately did not meet the requirements set for it to be further investigated in this study. This does not mean such efforts were in vain or should not be picked up by future researchers.

Other recommendations for future research include:

- Assessing the effect of varied sparging rates and sparging patterns, such as on-10 off-10, on-5 off-5 and continuously varied sparging rate in sync with or 90^{o} off from the production cycle such that maximum sparging occurs with minimum TMP or vice versa. As sparging consumes 30-70% of energy in AnMBR systems, optimization of this fouling management strategy is crucial [20], [75]. This research in general is still in its early stages and thus exploring it would be greatly beneficial.
- Acquiring a DC powered sparging pump that can be used with a VFD or directly controllable sparging pump, and modifying the current PLC with professional assistance to support sparging experiments, and a low cost backup/trial and error sparging pump such as the Win TC-100A 24 L lab pump, or their medical pump. As control ready, and DC VFD compatible dry vacuum pumps in the required size class are exceedingly rare, a better option may be to pursue an AC VFD with a rugged pump and additional professional assistance in electrical engineering.
- Increasing the regularity of solids sampling to twice weekly to limit interpolation required for presentation and modeling, while also more intentionally controlling the solids concentration to determine the effect of solids concentration on performance.

- Include DNA sampling at scheduled points in future operations as it is standard to include microbial community information with AnMBR studies regardless of the focus of the study.
- Continue efforts to model system performance and fouling based on observation.
- Thoroughly reexamine existing data produced by the system in an effort to gain insights that would be obscured in aggregate data, such as fouling behavior on the timescale of minutes and hours.
- Increasing the number of membrane modules used in the system or membrane surface area to provide greater control over production and HRT while operating within typical flux ranges.
- Consistently monitoring soluble COD and inorganics within the reactor liquor to better develop an understanding of potential foulants and foulant interactions.

These efforts have the potential to build on the findings of this research and improve on the quality of data collected. Future research on swine AnMBR operation and optimization has the opportunity to advance the platform to a more adoptable, societally beneficial, and economically viable technology. This research has hopefully provided some evidence of the value of such research and support for future work.

References

[1] M. H. Langholtz, B. J. Stokes, and L. M. Eaton, "2016 Billion-Ton Report: Advancing Domestic Resources for a Thriving Bioeconomy," DOE/EE-1440, ORNL/TM-2016/160, 1271651, Jul. 2016. doi: 10.2172/1271651.

[2] R. Cooper, "Beyond Waste: Sustainable Consumption for Community Resilience," in *The Community Resilience Reader: Essential Resources for an Era of Upheaval*, D. Lerch, Ed., Washington, DC: Island Press/Center for Resource Economics, 2017, pp. 261–278. doi: 10.5822/978-1-61091-861-9_16.

[3] "The-Sustainable-Development-Goals-Report-2022.pdf." Accessed: Dec. 27, 2023. [Online]. Available: https://unstats.un.org/sdgs/report/2022/The-Sustainable-Development-Goals-Report-2022.pdf

[4] M. Walsh, G. Schenk, and S. Schmidt, "Realising the circular phosphorus economy delivers for sustainable development goals," *npj Sustain. Agric.*, vol. 1, no. 1, Art. no. 1, Oct. 2023, doi: 10.1038/s44264-023-00002-0.

[5] G. Reta *et al.*, "Environmental impact of phosphate mining and beneficiation: review," *IJH*, vol. 2, no. 4, 2018, doi: 10.15406/ijh.2018.02.00106.

[6] Y. Wang *et al.*, "Mitigating Greenhouse Gas and Ammonia Emissions from Swine Manure Management: A System Analysis," *Environ. Sci. Technol.*, vol. 51, no. 8, pp. 4503–4511, Apr. 2017, doi: 10.1021/acs.est.6b06430.

[7] L. Zhang *et al.*, "A systematic review of life-cycle GHG emissions from intensive pig farming: Accounting and mitigation," *Science of The Total Environment*, vol. 907, p. 168112, Jan. 2024, doi: 10.1016/j.scitotenv.2023.168112.

[8] "us-ghg-inventory-2019-main-text.pdf." Accessed: Jan. 21, 2024. [Online]. Available: https://www.epa.gov/sites/default/files/2019-04/documents/us-ghg-inventory-2019-main-text.pdf

[9] J. N. Pretty, C. F. Mason, D. B. Nedwell, R. E. Hine, S. Leaf, and R. Dils, "Environmental Costs of Freshwater Eutrophication in England and Wales," *Environ. Sci. Technol.*, vol. 37, no. 2, pp. 201–208, Jan. 2003, doi: 10.1021/es020793k.

[10] C. Mainstone, R. M. Dils, and P. Withers, "Controlling Sediment and Phosphorus Transfer to Receiving Waters – A Strategic Management Perspective for England and Wales," *Journal of Hydrology*, vol. 350, pp. 131–143, Feb. 2008, doi: 10.1016/j.jhydrol.2007.10.035.

[11] K. a. Smith, A. j. Brewer, A. Dauven, and D. w. Wilson, "A survey of the production and use of animal manures in England and Wales. I. Pig manure," *Soil Use and Management*, vol. 16, no. 2, pp. 124–132, 2000, doi: 10.1111/j.1475-2743.2000.tb00187.x.

[12] K. a. Smith, A. j. Brewer, J. Crabb, and A. Dauven, "A survey of the production and use of animal manures in England and Wales. III. Cattle manures," *Soil Use and Management*, vol. 17, no. 2, pp. 77–87, 2001, doi: 10.1111/j.1475-2743.2001.tb00012.x.

[13] O. US EPA, "Anaerobic Digestion on Swine Farms." Accessed: Jan. 22, 2024. [Online]. Available: https://www.epa.gov/agstar/anaerobic-digestion-swine-farms

[14] I. Szűcs and V. Vida, "Global tendencies in pork meat - production, trade and consumption," *Applied Studies in Agribusiness and Commerce*, vol. 11, pp. 105–112, Dec. 2017, doi: 10.19041/APSTRACT/2017/3-4/15.

[15] P. M. Glibert, “From hogs to HABs: impacts of industrial farming in the US on nitrogen and phosphorus and greenhouse gas pollution,” *Biogeochemistry*, vol. 150, no. 2, pp. 139–180, Sep. 2020, doi: 10.1007/s10533-020-00691-6.
[16] A. Costa, C. Ely, M. Pennington, S. Rock, C. Staniec, and J. Turgeon, “Anaerobic Digestion and its Applications”.
[17] N. Key, W. D. McBride, M. Ribaudo, and S. Sneeringer, “Trends and Developments in Hog Manure Management:”.
[18] N. Scarlat, J.-F. Dallemand, and F. Fahl, “Biogas: Developments and perspectives in Europe,” *Renewable Energy*, vol. 129, pp. 457–472, Dec. 2018, doi: 10.1016/j.renene.2018.03.006.
[19] D. Bolzonella *et al.*, “Toward the Transition of Agricultural Anaerobic Digesters into Multiproduct Biorefineries,” *Processes*, vol. 11, no. 2, Art. no. 2, Feb. 2023, doi: 10.3390/pr11020415.
[20] K. Lim, “Anaerobic membrane bioreactors for domestic wastewater treatment: Treatment performance and fouling characterization,” 2021.
[21] U. W. R. Siagian *et al.*, “Performance and economic evaluation of a pilot scale embedded ends-free membrane bioreactor (EEF-MBR),” *Appl Microbiol Biotechnol*, vol. 107, no. 12, pp. 4079–4091, Jun. 2023, doi: 10.1007/s00253-023-12551-y.
[22] A. Al-Sayed, G. K. Hassan, M. T. Al-Shemy, and F. A. El-gohary, “Effect of organic loading rates on the performance of membrane bioreactor for wastewater treatment behaviours, fouling, and economic cost,” *Sci Rep*, vol. 13, no. 1, p. 15601, Sep. 2023, doi: 10.1038/s41598-023-42876-7.
[23] “membrane_bioreactor_fact_sheet_p100il7g.pdf.” Accessed: Dec. 27, 2023. [Online]. Available: https://www.epa.gov/sites/default/files/2019-08/documents/membrane_bioreactor_fact_sheet_p100il7g.pdf
[24] “Anaerobic Membrane Bioreactor (AnMBR) for Sustainable Wastewater Treatment ER-201434_Final_Report.pdf.” Accessed: Apr. 08, 2024. [Online]. Available: https://serdp-estcp-storage.s3.us-gov-west-1.amazonaws.com/s3fs-public/project_documents/ER-201434_Final_Report.pdf
[25] N. R. Buan, “Methanogens: pushing the boundaries of biology,” *Emerging Topics in Life Sciences*, vol. 2, no. 4, pp. 629–646, Dec. 2018, doi: 10.1042/ETLS20180031.
[26] W. E. Balch and J. G. Ferry, “Chapter One - The Wolfe cycle of carbon dioxide reduction to methane revisited and the Ralph Stoner Wolfe legacy at 100 years,” in *Advances in Microbial Physiology*, vol. 79, R. K. Poole and D. J. Kelly, Eds., Academic Press, 2021, pp. 1–23. doi: 10.1016/bs.ampbs.2021.07.003.
[27] G. Muyzer and A. J. M. Stams, “The ecology and biotechnology of sulphate-reducing bacteria,” *Nat Rev Microbiol*, vol. 6, no. 6, pp. 441–454, Jun. 2008, doi: 10.1038/nrmicro1892.
[28] S. Esquivel-Elizondo, P. Parameswaran, A. G. Delgado, J. Maldonado, B. E. Rittmann, and R. Krajmalnik-Brown, “Archaea and Bacteria Acclimate to High Total Ammonia in a Methanogenic Reactor Treating Swine Waste,” *Archaea*, vol. 2016, pp. 1–10, 2016, doi: 10.1155/2016/4089684.
[29] S. Z. Ahammad and T. R. Sreekrishnan, “Biogas: An Evolutionary Perspective in the Indian Context,” in *Green Fuels Technology: Biofuels*, C. R. Soccol, S. K. Brar, C. Faulds, and L. P. Ramos, Eds., Cham: Springer International Publishing, 2016, pp. 431–443. doi: 10.1007/978-3-319-30205-8_17.

[30] "Biogas Industry Market Snapshot | American Biogas Council." Accessed: Mar. 20, 2024. [Online]. Available: https://americanbiogascouncil.org/biogas-market-snapshot/
[31] Q. Chen and T. Liu, "Biogas system in rural China: Upgrading from decentralized to centralized?," *Renewable and Sustainable Energy Reviews*, vol. 78, pp. 933–944, Oct. 2017, doi: 10.1016/j.rser.2017.04.113.
[32] D. Thrän, K. Schaubach, S. Majer, and T. Horschig, "Governance of sustainability in the German biogas sector—adaptive management of the Renewable Energy Act between agriculture and the energy sector," *Energy, Sustainability and Society*, vol. 10, no. 1, p. 3, Jan. 2020, doi: 10.1186/s13705-019-0227-y.
[33] "WBA-Germany-4ppa4_.pdf." Accessed: Mar. 20, 2024. [Online]. Available: https://www.worldbiogasassociation.org/wp-content/uploads/2019/09/WBA-Germany-4ppa4_.pdf
[34] R. W. Field, D. Wu, J. A. Howell, and B. B. Gupta, "Critical flux concept for microfiltration fouling," *Journal of Membrane Science*, vol. 100, no. 3, pp. 259–272, Apr. 1995, doi: 10.1016/0376-7388(94)00265-Z.
[35] P. Bacchin, P. Aimar, and R. W. Field, "Critical and sustainable fluxes: Theory, experiments and applications," *Journal of Membrane Science*, vol. 281, no. 1, pp. 42–69, Sep. 2006, doi: 10.1016/j.memsci.2006.04.014.
[36] X. Shi, G. Tal, N. P. Hankins, and V. Gitis, "Fouling and cleaning of ultrafiltration membranes: A review," *Journal of Water Process Engineering*, vol. 1, pp. 121–138, Apr. 2014, doi: 10.1016/j.jwpe.2014.04.003.
[37] W. J. Gao, H. J. Lin, K. T. Leung, H. Schraft, and B. Q. Liao, "Structure of cake layer in a submerged anaerobic membrane bioreactor," *Journal of Membrane Science*, vol. 374, no. 1–2, pp. 110–120, May 2011, doi: 10.1016/j.memsci.2011.03.019.
[38] X.-M. Wang and X.-Y. Li, "Accumulation of biopolymer clusters in a submerged membrane bioreactor and its effect on membrane fouling," *Water Research*, vol. 42, no. 4–5, pp. 855–862, Feb. 2008, doi: 10.1016/j.watres.2007.08.031.
[39] H. J. Lin *et al.*, "Sludge properties and their effects on membrane fouling in submerged anaerobic membrane bioreactors (SAnMBRs)," *Water Research*, vol. 43, no. 15, pp. 3827–3837, Aug. 2009, doi: 10.1016/j.watres.2009.05.025.
[40] L. Zhou, B. Ye, and S. Xia, "Structural characteristics of cake layer in membrane bioreactor with chromate exposure," *Ecotoxicology and Environmental Safety*, vol. 169, pp. 583–589, Mar. 2019, doi: 10.1016/j.ecoenv.2018.11.056.
[41] X. Du, Y. Shi, V. Jegatheesan, and I. U. Haq, "A Review on the Mechanism, Impacts and Control Methods of Membrane Fouling in MBR System," *Membranes (Basel)*, vol. 10, no. 2, p. 24, Feb. 2020, doi: 10.3390/membranes10020024.
[42] N. AlSawaftah, W. Abuwatfa, N. Darwish, and G. Husseini, "A Comprehensive Review on Membrane Fouling: Mathematical Modelling, Prediction, Diagnosis, and Mitigation," *Water*, vol. 13, p. 1327, May 2021, doi: 10.3390/w13091327.
[43] H. Xu *et al.*, "A Simple Method to Identify the Dominant Fouling Mechanisms during Membrane Filtration Based on Piecewise Multiple Linear Regression," *Membranes (Basel)*, vol. 10, no. 8, p. 171, Jul. 2020, doi: 10.3390/membranes10080171.
[44] C. Duclos-Orsello, W. Li, and C.-C. Ho, "A three mechanism model to describe fouling of microfiltration membranes," *Journal of Membrane Science*, vol. 280, no. 1, pp. 856–866, Sep. 2006, doi: 10.1016/j.memsci.2006.03.005.

[45] Á. Robles *et al.*, "A review on anaerobic membrane bioreactors (AnMBRs) focused on modelling and control aspects," *Bioresource Technology*, vol. 270, pp. 612–626, Dec. 2018, doi: 10.1016/j.biortech.2018.09.049.
[46] R. A. Fox and D. C. Stuckey, "The effect of sparging rate on transmembrane pressure and critical flux in an AnMBR," *Journal of Environmental Management*, vol. 151, pp. 280–285, Mar. 2015, doi: 10.1016/j.jenvman.2014.08.011.
[47] Á. Robles *et al.*, "A review on anaerobic membrane bioreactors (AnMBRs) focused on modelling and control aspects," *Bioresource Technology*, vol. 270, pp. 612–626, Dec. 2018, doi: 10.1016/j.biortech.2018.09.049.
[48] H. Chang *et al.*, "Hydraulic irreversibility of ultrafiltration membrane fouling by humic acid: Effects of membrane properties and backwash water composition," *Journal of Membrane Science*, vol. 493, pp. 723–733, Nov. 2015, doi: 10.1016/j.memsci.2015.07.001.
[49] L. Wang, Z. Li, J. Fan, and Z. Han, "The intelligent prediction of membrane fouling during membrane filtration by mathematical models and artificial intelligence models," *Chemosphere*, vol. 349, p. 141031, Feb. 2024, doi: 10.1016/j.chemosphere.2023.141031.
[50] L.-F. Wang, D.-Q. He, W. Chen, and H.-Q. Yu, "Probing the roles of Ca2+ and Mg2+ in humic acids-induced ultrafiltration membrane fouling using an integrated approach," *Water Research*, vol. 81, pp. 325–332, Sep. 2015, doi: 10.1016/j.watres.2015.06.009.
[51] T. Lin, Z. Lu, and W. Chen, "Interaction mechanisms and predictions on membrane fouling in an ultrafiltration system, using the XDLVO approach," *Journal of Membrane Science*, vol. 461, pp. 49–58, Jul. 2014, doi: 10.1016/j.memsci.2014.03.022.
[52] O. US EPA, "Livestock Anaerobic Digester Database." Accessed: Mar. 29, 2024. [Online]. Available: https://www.epa.gov/agstar/livestock-anaerobic-digester-database
[53] P. J. Evans *et al.*, "A comparative pilot-scale evaluation of gas-sparged and granular activated carbon-fluidized anaerobic membrane bioreactors for domestic wastewater treatment," *Bioresource Technology*, vol. 288, p. 120949, Sep. 2019, doi: 10.1016/j.biortech.2019.01.072.
[54] X. Shi, G. Tal, N. Hankins, and V. Gitis, "Fouling and cleaning of ultrafiltration membranes: A review," *Journal of Water Process Engineering*, vol. 1, pp. 121–138, Apr. 2014, doi: 10.1016/j.jwpe.2014.04.003.
[55] X. Du, Y. Shi, V. Jegatheesan, and I. U. Haq, "A Review on the Mechanism, Impacts and Control Methods of Membrane Fouling in MBR System," *Membranes*, vol. 10, no. 2, Art. no. 2, Feb. 2020, doi: 10.3390/membranes10020024.
[56] Z. An *et al.*, "Anaerobic membrane bioreactor for the treatment of high-strength waste/wastewater: A critical review and update," *Chemical Engineering Journal*, vol. 470, p. 144322, Aug. 2023, doi: 10.1016/j.cej.2023.144322.
[57] C. V. Vedavyasan, "Chemical Cleaning of Membranes," in *Encyclopedia of Membranes*, E. Drioli and L. Giorno, Eds., Berlin, Heidelberg: Springer, 2015, pp. 1–8. doi: 10.1007/978-3-642-40872-4_633-4.
[58] A. Gul, J. Hruza, and F. Yalcinkaya, "Fouling and Chemical Cleaning of Microfiltration Membranes: A Mini-Review," *Polymers (Basel)*, vol. 13, no. 6, p. 846, Mar. 2021, doi: 10.3390/polym13060846.
[59] Office of Water, "Disinfectants and Disinfection Byproducts Rules (Stage 1 and Stage 2) What Do They Mean to You?," *EPA 815-R-20-005*.
[60] J. Martin, "An Evaluation of a Covered Anaerobic Lagoon for Flushed Dairy Cattle Manure Stabilization and Biogas Production," *EPA Contract No. GS-10F-0036K*, Jun. 2008.

[61] C. Feng, S. Shimada, Z. Zhang, and T. Maekawa, “A pilot plant two-phase anaerobic digestion system for bioenergy recovery from swine wastes and garbage,” *Waste Management*, vol. 28, no. 10, pp. 1827–1834, Jan. 2008, doi: 10.1016/j.wasman.2007.08.009.

[62] C. Shin, P. L. McCarty, J. Kim, and J. Bae, “Pilot-scale temperate-climate treatment of domestic wastewater with a staged anaerobic fluidized membrane bioreactor (SAF-MBR),” *Bioresource Technology*, vol. 159, pp. 95–103, May 2014, doi: 10.1016/j.biortech.2014.02.060.

[63] F. Bu, S. Du, L. Xie, R. Cao, and Q. Zhou, “Swine manure treatment by anaerobic membrane bioreactor with carbon, nitrogen and phosphorus recovery,” *Water Science and Technology*, vol. 76, no. 8, pp. 1939–1949, Oct. 2017, doi: 10.2166/wst.2017.278.

[64] S. I. Padmasiri, J. Zhang, M. Fitch, B. Norddahl, E. Morgenroth, and L. Raskin, “Methanogenic population dynamics and performance of an anaerobic membrane bioreactor (AnMBR) treating swine manure under high shear conditions,” *Water Research*, vol. 41, no. 1, pp. 134–144, Jan. 2007, doi: 10.1016/j.watres.2006.09.021.

[65] H. Chen *et al.*, “Swine wastewater treatment using combined up-flow anaerobic sludge blanket and anaerobic membrane bioreactor: Performance and microbial community diversity,” *Bioresource Technology*, vol. 373, p. 128606, Apr. 2023, doi: 10.1016/j.biortech.2023.128606.

[66] J. Tang *et al.*, “Enhanced methane production coupled with livestock wastewater treatment using anaerobic membrane bioreactor: Performance and membrane filtration properties,” *Bioresource Technology*, vol. 345, p. 126470, Feb. 2022, doi: 10.1016/j.biortech.2021.126470.

[67] T. Inaba, T. Hori, R. R. Navarro, A. Ogata, D. Hanajima, and H. Habe, “Revealing sludge and biofilm microbiomes in membrane bioreactor treating piggery wastewater by non-destructive microscopy and 16S rRNA gene sequencing,” *Chemical Engineering Journal*, vol. 331, pp. 75–83, Jan. 2018, doi: 10.1016/j.cej.2017.08.095.

[68] “7-iSep500UFProductManual.pdf.” Accessed: Mar. 02, 2024. [Online]. Available: https://d3pcsg2wjq9izr.cloudfront.net/files/11144/download/423902/7-iSep500UFProductManual.pdf

[69] “Instruction_Manual_HSU_201403.pdf.” Accessed: Mar. 02, 2024. [Online]. Available: https://www.water.toray/knowledge/manual/pdf/Instruction_Manual_HSU_201403.pdf

[70] W. Yang, N. Cicek, and J. Ilg, “State-of-the-art of membrane bioreactors: Worldwide research and commercial applications in North America,” *Journal of Membrane Science*, vol. 270, no. 1, pp. 201–211, Feb. 2006, doi: 10.1016/j.memsci.2005.07.010.

[71] S. Xia *et al.*, “The effect of organic loading on bacterial community composition of membrane biofilms in a submerged polyvinyl chloride membrane bioreactor,” *Bioresource Technology*, vol. 101, no. 17, pp. 6601–6609, Sep. 2010, doi: 10.1016/j.biortech.2010.03.082.

[72] “Anaerobic Digestion: Biogas Production and Odor Reduction.” Accessed: Mar. 20, 2024. [Online]. Available: https://extension.psu.edu/anaerobic-digestion-biogas-production-and-odor-reduction

[73] N. Chen *et al.*, “Advancements in swine wastewater treatment: Removal mechanisms, influential factors, and optimization strategies,” *Journal of Water Process Engineering*, vol. 54, p. 103986, Aug. 2023, doi: 10.1016/j.jwpe.2023.103986.

[74] L. Wang, Z. Li, J. Fan, and Z. Han, "The intelligent prediction of membrane fouling during membrane filtration by mathematical models and artificial intelligence models," *Chemosphere*, vol. 349, p. 141031, Feb. 2024, doi: 10.1016/j.chemosphere.2023.141031.

[75] Z. Liu *et al.*, "Quantitative relationships for the impact of gas sparging conditions on membrane fouling in anaerobic membrane bioreactor," *Journal of Cleaner Production*, vol. 276, p. 123139, Dec. 2020, doi: 10.1016/j.jclepro.2020.123139.

Appendix A - Correlation of Parameters from Phase I

	Test t					
	X1	X2	X3	X4	X5	X6
X1	1					
X2	0.258333	1				
X3	-0.70699	-0.44123	1			
X4	0.043616	0.00558	-0.11166	1		
X5	0.357744	0.152296	-0.23833	0.27905	1	
X6	-0.04042	0.2340	-0.12117	0.049797	-0.01419	1
	Pearson r					
	p value (p<0.1)					
X1		0.134	2.06E-06	0.8035	0.03486	0.8177
X2			0.007973	0.9746	0.3825	0.1761
X3				0.5231	0.168	0.4881
X4					0.1045	0.7763
X5						0.9355
X6						

X1: CODin (mg)

X2: Solids Rejection (g/m^2)

X3: Avg HRT (Days)

X4: Cleaning Frequency (# of cleans)

X5: VS/TS

X6: pH

Data points used in this comparison were from daily summaries.

Appendix B - Appendix B – Linearity of Fouling Model With Respect to Fouling Data