University of Alberta

Evaluation of an Immersed Hollow Fibre Membrane Technology for the Treatment of Swine Waste Supernatant

by

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requirements for the degree of Master of Science

in

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LIST OF SYMBOLS, NOMENCLATURE OR ABBREVIATIONS

Symbols

J ₂₀	Normalized specific flux (L h ⁻¹ m ⁻² kPa ⁻¹ @20°C)	
IL	Permeate (In-Line or composite sample as identified)	
РТ	Process tank	
<u>Abbreviations</u>		
ASP	Activated sludge process	
CWQG	Canadian Water Quality Guidelines	
CFO	Confined feeding operation	
MBR	Membrane bioreactor	
MLSS	Mixed liquor suspended solids	
Standard Methods	Standard Methods for the Examination of Water and Wastewater (APHA 1999)	
SWSN	Swine waste supernatant	
UFAF	Up-flow anaerobic filter	
<u>Nomenclature</u>		
ZW-1 testing	Compact Zeeweed [®] 500 membrane module for bench top	
ZW ₁ D ₂₀ F ₅₀	means #1 ZW-1 module used at a dilution of 20% SWSN and an initial flowrate of 50 mL/min	

1. INTRODUCTION

Limited water availability is an impediment to the development of the agricultural industry in Alberta especially with the increase in the size of modern intensive livestock operations. Water reuse is one option to reduce the quantity of raw water supply required and promote the advantages of self-sufficiency and closed systems. Increased sustainable agricultural production in Alberta benefits the economy of Alberta and livelihood of its farming residents.

Properties that are desired in a water or wastewater treatment system for rural water reuse are as follows:

- scalability especially in the low capacity range;
- low or no dependence on chemical additives and dosing; and
- flexibility to integrate into existing operations or with other easily accessible technologies.

The agricultural industry in Canada produces low cost crops and meat for value added processing or exports and has not significantly supported the research or interest of water treatment or reuse technologies. Because of this, adaptive research from technologies produced for other industries is a common trend and obstacle. In general, the new membrane technologies share many of the properties looked for in these applications and deserve investigation. In particular the relatively new immersed hollow fiber membranes may provide the degree of flexibility and operating characteristics needed. A current investigation into agricultural wastewaters from intensive livestock operations and the issues surrounding their reuse is required to define and target cost-effective, workable treatment technologies. This work is to serve as a springboard to evaluate promising treatment technologies, and in this project specifically, the relatively new immersed membrane systems. A workable reuse technology could reduce the raw water requirement of larger confined feeding operations (CFO) as well as the operating costs surrounding water access and use. Water/wastewater systems, which more closely approach closed systems in regard to water, have more opportunity available for development location, acceptance, and sustainability

1.1 Swine Production

In Canada, swine are almost always housed indoors in separate areas according to their age and physiological state where temperature and feed can be controlled to optimize production levels. Manure is usually handled in a liquid form upwards of 90% moisture and changes little from this moisture state until removal to storage reservoirs. Due to behaviors of swine significant water above physiological requirements can be spilled and become part of the waste load. Pigs have been known to waste up to 20 litres of water per day per head and new wet feed, water system and water nipple designs are countering some of this wastage (Phillips, et al 1995). Unlike beef cattle, swine are produced in a wide variety of climatic regions. Wastes stored in anaerobic lagoons or uncovered storage in Alberta is subject to rainfall and snowmelt, hoever, in most locations evaporation exceeds precipitation. In Alberta, livestock wastes from swine operations are mainly stored in earthen manure storages and the liquid manure undergoes little treatment. Although the liquid manure and accompanying wastewaters are referred to as swine wastes in this project, the "wastes" generated can provide a valuable nutrient and organic matter resource to cropland when applied at appropriate rates. However, where microbial concerns are present, they must be addressed. Wastes are mixed prior to land application but before this a supernatant can be identified. As to condition in lagoons, the material varies from a less concentrated light yellow green to a black more concentrated form (U.S. Soil Conservation Service 1992). It is this swine waste supernatant (SWSN) that will be targeted for treatment and potential reuse. Both open gutter and below-slat

flushing systems exist for transporting wastes out of the barn and can add significantly to the wastewater quantity production.

According to published values, the amount of water used and waste volume and characteristics can vary significantly among swine operations. Water is used for drinking, cooling, washing, and domestic needs with the largest percentage being for drinking. A manure production and characteristics standard has been used to assist in the planning, design, and operation of manure collection, storage, pretreatment and utilization systems for livestock enterprises (American Society of Agricultural Engineers 2003). Table 1 provides the values for fresh (as voided) faeces and urine which are calculated from data combined as an average from a wide base of published and unpublished data. In lieu of data, actual samples should be used since values can change with animal diet, age, usage, productivity and management.

1.2 Membrane Treatment of Wastewater

The use of membranes in the treatment of wastewater is evolving from membrane used in a direct filtration mode to the more recent systems, which couple them with bioreactors of various configurations.

Agricultural wastewaters have high carbon and high nutrient contents and therefore biological treatment to reduce this organic loading is indicated. The key to this is to produce and retain an actively growing biomass in the reaction vessel. Whether suspended or attached this biomass requires oxygen to avoid going anoxic and often requires a significant energy outlay to provide air required. Submerged or immersed membranes have allowed the coupling of this biological process with physical/chemical treatments within the reaction vessel. Two municipal wastewater treatment processes, which perform this, are the membrane-coupled activated sludge process (MCASP) and the membrane bioreactor (MBR).

The conventional activated sludge process has served the needs of municipal wastewater treatment for decades since its development in England (Adern and Locket 1914).

Beyond a straight aeration process, the activated sludge process (ASP) retains sludge with its mix of living organisms to separate the wastes from the water by its conversion to biomass and sedimentation into a sludge blanket and a relatively clear supernatant. The maintenance and recycling of this activated sludge is key to optimum performance of this process. Although the topic of ASP will not be discussed further here, the MCASP has emerged to deal with some limitations of ASP. In conventional ASP a final clarifier only retains the activated sludge that forms flocs and settles (Günder 2001). With membrane filtration, all parts of the activated sludge that are larger than the cutoff of the membrane are retained. As a result, the separation of the activated sludge from the cleaner wastewater is independent of the sedimentation qualities of the activated sludge and is only dependent on the inserted membrane.

Membrane bioreactors (MBR) utilize the membrane as a solid separation device in the activated sludge aeration. Filtration capacity can be altered by increasing the depth of the cake layer which can form on the membrane. Secondly, the maintenance of the membrane and the removal of the biofilm is a major component of the system design.

Efforts to produce a compact membrane bioreactor system started in the early 1970" with the introduction of the Cycle-Let[®] system (Cote and Thompson 2000). In the late 1980's a shell-less membrane module, suitable for immersion directly in the biomass evolved and became the product Zeeweed[®] used in the ZenoGem[®] process. The key features to this membrane were low vacuum pressure, outside-in permeate flow, air bubble scouring of the hollow fibres, and modular-scalable design. This allowed for simplicity and a reduction in energy costs for filtration. A progression of improvements to design evolved with a key one being packing density of the cassettes. The Zeeweed[®] ZW-500 membrane, on which this project is based, has a packing density of 146 m²/m³ for the standard 8 module cassette. The ZW-1 membrane module can be used for preliminary investigation into the performance of commercial ZeeWeed[®] 500 modules. Zenon states the results would better represent effluent quality than fouling behavior due to the limitations of the ZW-1's compact design (Zenon Environmental Enterprises Ltd. 2001).

1.3 Research Objectives

The main focus of this study was to evaluate a low pressure immersed hollow fibre membrane in its ability to reduce key contaminants present in swine wastewater. To do this, the following steps were required and will be focused on in this document.

- Develop and/or refine a methodology and bench top data acquisition and control system suitable for operating a low pressure immersed hollow fibre membrane module in direct filtration mode in polluted water.
- Establish the maximum sustainable flux capability to produce 10 L of permeate.
- Determine the membrane's ability to reduce key contaminants in the supernatant of swine wastes at this level of flux.
- Make a preliminary assessment of the membrane size required and its potential for treatment of swine wastewater in the context of current swine production systems.

2. LITERATURE REVIEW

2.1 Production and Characteristics of Swine Manure

The level of production and characteristics of swine manure can vary significantly among herd production and waste management systems. Table 1 is adapted from a North American standard for livestock manures to assist in the design of these systems (American Society of Agricultural Engineers 2003). Facility specific sampling and analysis should be used when possible.

A more recent field investigation of water usage and manure production rates used by the swine industry and regulative authorities is available as a non-peer reviewed source and is compared to monitoring results from a cross section of herd sizes (Froese 2003). Nine hog operations in Manitoba and Saskatchewan were enlisted in the study supported by the Manitoba Livestock Manure Management Initiative (MLMMI) but only four of the operations could provide reliable waste production data. The two largest herds had the lowest daily per head waste production rates and averaged 7.9 L/day/pig (7.1 to 9.1) for grower/finisher herds. It is evident in this type of study that reliable data from cooperators of active operations may be an issue. The waste production values for grower finisher were compared to other published values from the Prairie Swine Center (Saskatoon, SK), USA, and The Netherlands at 8.5, 4.5, and 3.1 L/day/pig, respectively. The spread of values indicates that comparisons of economics generated from condensed measurements (i.e L/pig/day) should be done so cautiously.

A spreadsheet calculator produced by the author, based on Canadian swine management guidelines, was available for estimates of animal populations, water requirements and waste production based on inputs of herd makeup and performance.

Parameter	Unit	Mean *	Std. Dev.
Total manure	kg	84	24
Urine	kg	39	4.8
Density	kg/m ³	990	24
Total Solids	kg	11	6.3
Volatile Solids	kg	8.5	0.66
BOD ₅	kg	3.1	0.72
COD	kg	8.4	3.7
pH	kg	7.5	0.57
TKN	kg	0.52	0.21
Ammonia N	kg	0.29	0.1
Total Phosphorus	kg	0.18	0.1
Orthophosphorus	kg	0.12	n/a
Potassium	kg	0.29	0.16
Calcium	kg	0.33	0.18
Magnesium	kg	0.07	0.035
Sulfur	kg	0.076	0.04
Sodium	kg	0.067	0.052
Chloride	kg	0.026	0.052
Iron	kg	16	9.7
Manganese	kg	1.9	0.74
Boron	g	3.1	0.95
Molybdenum	g	0.028	0.03
Zinc	g	5	2.5
Copper	g	1.2	0.84
Cadmium	g	0.027	0.028
Nickel	g	n/a	n/a
Lead	g	0.084	0.012
Total coliform bacteria	colonies (10^{10})	45	33
Fecal coliform bacteria	colonies (10^{10})	18	12
Fecal streptococcus bacteria	colonies (10^{10})	530	290

Table 1Fresh Swine Manure Production and Characteristics per 1000 kg Live Animal
Mass per Day (adapted from ASAE 2003)

Based on typical swine animal mass of 61 kg

Feces and urine as voided

All nutrients and metal values given in elemental form

* Mean based on 58 data points

2.2 Treatment of Livestock Wastes

Waste treatment in an agricultural sense differs from the typical way we think of the treatment of industrial or municipal wastes. It largely has to do with the scale of the operation. The basic asset and difference is land. If an enlarged spatial view greater than

the cropped land base is examined, then modifications of the natural environment for the treatment of dilute agricultural wastes are possible. Vegetative filter strips, riparian buffers, natural wetlands fall into this category and have received mixed acceptability to the public or regulative agencies.

Agricultural wastes are very high in organic content, which make biological processes attractive to utilize and reduce this material. Soil applied livestock wastes, sometimes termed organic fertilizer, can actually enhance the land ecosystem in soils with low carbon content or fertility. With a sufficient land base available, the "treatments" most commonly applied, are not treatments in the normal sense. The structures or management practices are more targeted at the following:

- store the wastes generated to allow for land application at a time which crops and soil can safely assimilate them;
- stabilize the wastes generated so as not to produce excessive objectionable odours during storage or spreading; and
- mixing of the wastes to reduce the variability in nutrient and consistency making the results from land application more consistent.

The basic biological processes available that function with varying success are, by microorganism type - aerobic, anaerobic, facultative, and photosynthetic, or by process type suspended growth and attached growth. Basic equations have been developed for a completely mixed biological treatment process. With the solids content and particle size present in agricultural wastes, complete mixing assumptions must be checked closely (Loehr 1984). Loehr goes on to provide many insights into the differences between municipal and agricultural wastes. For lagoons to achieve an aerobic state, very large areas would be required. As a result anaerobic ponds are more commonly used. Many lagoons currently called anaerobic are actually overloaded aerobic lagoons. The effectiveness of anaerobic degradation is temperature dependent so designs that retain heat in cold climates should be more effective.

When evaluating wastewater treatment processes against traditional waste management practices the full costs should be evaluated. Wastewater treatment while seen as a new cost can produce savings in labor, equipment and land for spreading, and odour reductions.

Since so many treatment options, regulative requirements, and applications abound water and wastewater treatment a means of managing this knowledge is of great benefit to the many stakeholders. Aside from the traditional literature searches available through libraries, an electronic database capable of collecting and searching the current and emerging technological information is useful and has been developed. This task was undertaken by the Canadian Water and Wastewater Association (CWWA) and its network and made available the spring of 2002 on their website (CWWA 2004). A researcher can find relevant information organized by substance removed or the technology employed. This database including 528 treatment descriptions was searched and produced 18 applicable to agricultural wastes. However, none of the 18 was identified as a reuse or reclamation technology and the use was "discharge to the environment". Many dealt with odour reduction or methods of reducing the size of lagoon storage. This site is under development but promises to be a substantial tool. A database for legislation pertaining to de-centralized or on-site systems is under development and a database specifically targeting agriculture is proposed.

The potential of closed water systems on dairy farms was studied and determined that the most plausible options for closed water systems on dairy farms was the collection and use of rain water and treatment and reuse of wastewater for irrigation, manure flushing and animal drinking water (Willers, et al 1999). It was left as a need for further research whether the effluents could be reclaimed to the safety level required for animal drinking water. In looking at the issue in the study area of the Netherlands, Greece and North America it stated that it is prohibited that water that has been in contact with manure or human waste be used for milking related cleaning. It stated that sophisticated treatments like membrane filtration and UV disinfection are still very costly, making small-scale implementation on dairy farms not economically feasible. The risk of pathogen breakthrough and disease spreading by reuse of such effluents needed to be quantified.

If the end use of treated wastewater is as a hydraulic transport medium (i.e. flushing gutter), treatment options are less complex and more diverse. Examples of research in this area date back much longer as discussed in three biological swine waste systems with these flush systems were evaluated including:

- aerobic basin 28 sow farrowing barn;
- anaerobic lagoon/ aeration basin 56 sow farrowing barn; and
- anaerobic lagoon/rotating biological contactor (RBC) 700 hog feeder operation in four barns (EPA 1974).

All of these units can be considered very small by today's industry standard but it points out that even when successful in achieving a high organic matter removal rate other assessments will prevail for acceptability. Even though odours were low in the buildings and similar between treatments, the aerobically treated wastewater was more desirable to humans present. Mineral crystallization on the RBC unit was observed and would have to be periodically removed with a dilute acid. The RBC unit of the time was deemed not to be a suitable piece of equipment for the conditions present in intensive livestock operations. Overall the anaerobic lagoon/ aerobic basin was most desirable in terms of operational difficulty and cost. In terms of BOD₅, COD, total solids, volatile solids and phosphates the removal rates were 92, 96, 75, 94, and 0% respectively. This evaluation is quite dated now.

A continuous flow anoxic/aerobic (A/A) biological treatment system designed to treat swine wastewater was investigated (Pan and Drapcho 2001). Laboratory studies were conducted to determine the heterotrophic bacteria kinetic parameter values in swine wastewater. Maximum growth rates at 20 °C were low, 0.075 and 0.055 per hour for aerobic and anoxic conditions, respectively, indicating that swine wastewater is relatively difficult to degrade. A bench–scale A/A system operating at 35 and 36 hours hydraulic retention time and a recirculation ratio of 1.0 showed that it could be effective in reducing the concentration of organic compounds, inorganic nitrogen and sulfide in swine wastewater. In a recent investigation, physical/chemical treatments were applied to swine wastewater from the Swine Research and Technology Center located at the University of Alberta Edmonton Research Station (Zhu 2003). In the lead up study to evaluating a customized system, alum was found to be the most effective coagulant in reducing both total suspended solids (TSS) and total phosphorus (TP). The customized design of the physical/chemical treatment system included an upflow sludge blanket clarifier followed by patented Martin filters (upflow fluidized bed filters). The major objective of this project was to investigate the best operating conditions for swine wastewater treatment by physical/chemical processes at the bench and field pilot scale. This investigation is still underway but the work completed to date provided background information to the characterization and analysis of the swine wastewater used. At an alum dosage of 1,600 mg/L the removal efficiency of TSS in the pilot and laboratory test was 55% and 54%, respectively. Another parameter achieving significant removal was TP at 69 % and 68%, respectively, for the same run.

There is a cost to treating swine wastewater to a level acceptable for release to a water body as is commonly done with the treatment of municipal effluents. Recouping the value of treated wastewater through reuse is becoming an increasingly attractive proposition. The idea of treating swine wastewater or liquid manure, which is greater than 90% water as a source for drinking water for pigs, is one scenario. (Navaratnasamy 2003). Removal of pathogens (<5,000/100 mL) and reduction of TDS (< 3,000 mg/L) were the key targets investigated in this study. Aeration reduced TDS levels at a faster rate than ozone treatment or natural settling. Slow sand filtration reduced pathogens in an aerated liquid to meet this recommended level of for swine drinking water. By these criteria, diluting with 20% fresh water with slow sand filtered liquid after 7 days of aeration would be sufficient. This was at an estimated energy cost of \$0.20/growing pig.

An entrapped mixed microbial cells (EMMC) process was used to investigate the simultaneous removal of carbon and nitrogen from dilute swine wastewater (Yang, et al 2003). Of all the pretreatment and post treatment combinations tried with this process an ammonium crystallization pretreatment and lime post treatment produced the best removal efficiencies (over 30 days) of carbon, nitrogen and phosphorus. The EMMC

process alone removed 83.5% total chemical oxidation demand (TCOD) and 95% TN following pretreatment. Lime post treatment coupled with this process removed 98% of TP. The dilute swine wastewater from a 2,000 pig operation produced the best estimated treatment costs of \$4.91US/pig/year. This prototype study, which used anaerobic lagoon treated wastewater as its source, was performed in Hawaii and would have to be replicated for harsh Alberta winters and economics existing in livestock production.

The performance of a pilot-scale upflow anaerobic filter (UFAF) treating a dairy wastewater was studied for a period of 3 months (Ince 1998). The UFAF achieved more than 85% COD and 90% BOD₅ removal efficiencies at a loading rated of 6 kg COD/m³.d with an HRT of 20 hours.

The literature review produced few projects that claimed or suggested that agricultural wastewaters could or should be taken to the point of recycling reclaimed water to animal watering.

2.3 Membrane Treatment

Membranes can be constructed in many configurations and levels of tightness to achieve the targeted separation of the constituents of a solution found in various applications. Unlike a filter, which uses physical straining, to separate particulate from the solution a membrane can use size, shape, and charge to separate selectively and to a finer degree.

The transportation of constituents in a solution can occur through the pores by convection or through the membrane material itself by diffusion. Hagen and Poiseuille laws describe the flow through pores by convection and Ficks law describes the diffusion processes. Membranes achieving micro or ultrafiltration are sometimes called pore membranes and fall into the former transport mode. Nanofiltration or reverse osmosis membranes are sometimes called solubility membranes due to the latter transport mode. Figure 1 illustrates the classification of pressure-driven membrane processes and common drinking water pathogens by their size and molecular weight range (EPA 2003a). Hollow tube or hollow fibre membranes are one configuration that has been developed to deal with some of the fouling and maintenance problems that exist. Flow of the permeate can be from inside out or from outside in. It is this latter configuration that has been investigated in this project. With membranes the main factors affecting the volume of water moving across the membranes are the differential pressure, the flux, the membrane area, and the temperature of solutions.



Figure 1 Filtration Application Guide for Pathogen Removal (adapted from EPA 2003a)

With the advantages of scalability and positive pathogen barriers that membrane technologies can provide, there is interest in making them work for a wide number of applications. Ultrafiltration of agricultural wastewaters with organic and inorganic membranes was studied in Germany largely due to the potential that the inorganic membranes have in mechanical, chemical, and thermal persistence (Reimann and Yeo 1997). Wash waters from potatoes and carrots, pig and cattle slurry and milk plants were tested under constant conditions for pressure, temperature, and COD concentration in the feed water. Of those tested a silicon carbon inorganic membrane with a cutoff of 0.05 μ m produced the greatest permeate flux and best COD rejection at 79 and 72% for pig and cattle slurry, respectively. Reimann and Yeo (1997) found that the optimum transmembrane pressure amounted to 200 kPa with a higher pressure not leading to a

proportional increase in permeate flux. In this process, control of the optimum pressure must be determined experimentally.

A lab-scale integrated biological treatment and membrane separation system was studied for treatment of swine wastewater to achieve energy recovery, fertilizer, production and water reclamation (Zhang, et al 2002). The effective treatment train evaluated in the experimental set up consisted of the following:

- anaerobic sequencing batch reactor 12 L;
- two aerobic sequencing batch reactors 4L and 7.5 L;
- a sand filter 4 grain sizes, 0.61 m total depth; and
- a RO membrane, spiral-wound operating at 10% recovery of water (permeate).

The membrane separation showed in a batch treatment there are diminishing returns to permeate production in terms of total nitrogen. When the process tank volume was reduced to 50%, 90% of the total nitrogen was in the concentrate, but when it was reduced to 10% of original volume, 70% of original total nitrogen was in the concentrate.

Membrane treatment of livestock wastes to reduce volume has been investigated in Europe to potentially reduce the cost of storage, transportation and spreading without significant losses of nitrogen and phosphorus compounds. Treatment of liquid effluents from dairy cattle and pigs using reverse osmosis (RO) was studied in Sweden (Thorneby, et al 1999). Since losses of ammonia to the atmosphere are also a concern, improved separation of faeces and urine, less delay in tank storage of excreted manure, and improved land spreading techniques are important. While greater concentration of nutrients may be seen as a problem in a municipal sense, it is often a problem of low and/or imbalanced nutrient concentration hindering the economics of land spreading organic wastes as a fertilizer. Reducing dilution is the first step but concentration of wastes by volume reduction or dewatering was the idea driving the research of RO. While it was possible to reduce the volume of the liquid fraction of slurry by up to 60%, fouling problems caused a significant flux reduction in pure water flux measured after the waste treatment. Retention of environmentally hazardous substances was greater than 98% except ammonia in the 94 to 98% range. While raising temperature of the effluent will improve and lower unit membrane costs, odour and volatilization losses of ammonia will increase.

In many studies reviewed a pretreatment of swine wastes by natural settling was reported as comparable or superior and the evaluation focused on what to do next to these organically rich effluent (DeKleijn and Voermans 1991). A volumetric waste stream reduction of 77% was obtained from the a process sequence of sieving, microfiltration and reverse osmosis (Pieters, et al 1999). Increasing the temperature of the liquid prior to microfiltration gave rise to higher fluxes. A mean flux of 159.1 L h⁻¹m⁻² was determined for a ceramic membrane (Sephi-matic 184R) for sow slurry sieved through a 100 µm bag filter. The capital, maintenance, and power costs for a system capable of treating 1100 sows was estimated at \$8 U.S per m³ of raw sow slurry. During the experiments, membranes were cleaned by back-pulsing the microfiltrate or permeate at regular intervals (5 second duration at 229.5 sec intervals). Raw waste characteristics, pretreatments, types of membranes, and applied pressures will all influence the appropriate cycle duration and cleaning routine.

Concern over changing global climate has been heightened in many regions where severe drought has occurred in recent years. During these periods, small surface water supplies often dry up and groundwater reserves are more heavily impacted. Where economies are heavily reliant on agriculture, treated municipal wastewater is targeted for reuse for irrigation of crops. As an example of this, water from a secondary municipal treatment in Southern Italy underwent tertiary treatment with a membrane pilot plant which incorporated Zenon hollow fibre technology (Pollice, et al 2004). This water was to be used for agricultural irrigation on tomato and fennel crops so microbiological quality was important as well as the effect on the soil. As expected, soluble nitrogen and phosphorus passed through the membrane but this was not a concern as this as a beneficial crop fertilizer when balanced with crop needs. Performance in suspended solids and bacterial removal were deemed very good and produced better quality water than the well water it

was compared with. However, results did not show an absolute barrier to bacteria and potential pathogens as would be expected from the 0.03 µm pore size but was subjectively explained by the likely contamination through pump and valves connected to municipal wastewater. Negative microbiological contamination of crops or degradation of soil was not indicated in this two year study.

The ZeeWeed[®] ZW-500 membrane filtration system has been evaluated by the National Sanitation Foundation (NSF) in the Environmental Technologies Program created by the U.S. Environmental Protection Agency (EPA) (EPA/NSF 2001). In three test periods, each a minimum of 30 days long, turbidity was decreased from > 200 NTU to < 0.05 NTU, 95% of the time. Three to four log removal was obtained for particles > 2 microns and a 4, 3 and 3 log removal was obtained for *Cryptosporidium*, viruses and *Giardia*, respectively. The permeate flux exceeded 76.4 L h⁻¹m⁻² (normalized to 20°C). At the 38.6 kPa operated at during the first test period this converts to a specific flux of 1.98 L h⁻¹m⁻² kPa⁻¹ @20°C. Feed water was much cleaner than the diluted swine waste supernatant treated in this project except during challenge tests in some parameters.

2.4 Regulatory Considerations for Wastewater Reuse

Repeated reference is made in agricultural codes of practice that water should be potable or "suitable for drinking". Some direction as to what potable is may be taken from the Canadian Water Quality Guidelines (CWQG). In agricultural production there is a relaxation of some water quality guidelines compared to human health and an opportunity exists for utilizing water of less quality, but not at the expense of animal health or consumer confidence and acceptance. CWQG identifies some of the production parameters but since chronic exposure is not as often an issue there may be room to maximize the efficiency of water use.

The overriding statement relevant to Canadians pertinent to water reuse is in Section 7 of the National Plumbing Code of Canada (Canadian Commission on Building and Fire Codes 1995) states simply that "a non-potable water system shall not be connected to a potable water system". Non-potable water piping shall be identified by markings that are permanent, distinct, and easily recognized. Where water reuse has been promoted a

separate line of distinct purple piping is often used. Unless wastewater treatment can assure safe, potable water this requirement may become a factor in the cost of a new system. Education and awareness of all users of a non-potable water supply should go hand in hand with the adoption of these enabling technologies.

The Unites States is the largest importer of livestock production from Canada. If we look to the US for the eventual promulgation of our legislation or system standards, the recent requirements of the Long Term 2 Enhanced Surface Water Treatment Rule (LT2ESWTR) contains guidelines on applying membrane filtration to meet these requirements (EPA 2003b). Microfiltration and ultrafiltration processes that meet this rule will receive *Cryptosporidium* spp. removal credits. Although these processes have significantly advanced the water treatment technology "toolbox", the weak point of membrane processes is ensuring the integrity of the membrane. Systems that can demonstrate this integrity before installation and in ongoing daily tests are also eligible for 2.5 log additional credit for *Cryptosporidium* spp. removal credits.

Liquid swine waste typically contains up to 5% solids and might be seen as a feed source if some treated portion of it is used for animal watering. Since a significant component of animal waste is protein, fibre, and essential minerals still available for metabolism there may be a tie to the practice of recycling animal waste generated from slaughter into some animal feeds. Since this is seen as a local practice more effectively regulated by the state the FDA has lessened its Federal regulatory control but has not endorsed this practice either (FDA 1995). The Association of American Feed Control Officials carries the regulatory arm at the State level and follows their "Model Regulations for Processed Animal Waste Products as Animal Feed Ingredients". With the recent impact of Bovine Spongiform Encephalopathy (BSE or Mad Cow disease) these regulations are becoming stricter and no doubt more stringently enforced.

In Canada, the Feed Act enforced by the Canadian Food Inspection Agency covers the same regulative ground. It specifically exempts feeds if it is manufactured by a livestock producer, if it is not offered for sale and has not had incorporated into it any drug or other substance that may adversely affect human health or the environment (Canadian Food

Inspection Agency 1983). Other than the applications of the plumbing code, the on-farm measure that would most influence on-farm water reuse is a voluntary program of hazard analysis currently being provided. The on-farm food safety programs based on the internationally accepted Hazard Analysis Critical Control Point (HACCP) system may have an influence on the uptake of wastewater treatment and reuse technologies.

2.5 Risk Management

2.5.1 Laboratory and Bench Top Testing

Swine wastes may contain a number of pathogenic organisms or their products. A "nonexhaustive" list containing known infectious substances that can be present in swine wastes is shown in Table 2 and formed part of the material safety data manual for the laboratory investigation (Health Canada. 2004). More detail on their health hazard is provided online at the Health Canada website.

Biohazard safety Level 2 practices are required in working with swine wastewater in this laboratory investigation. These include the use of gloves and protective wear, avoidance of breathing in aerosols, and use of chemical disinfectants. The common practice cited and used to deal with small spills was:

- allow aerosols to settle and wear protective clothing;
- gently cover with paper towels and apply 1% sodium hypochlorite, starting at perimeter and working towards the center. Allow sufficient time before clean up (30 minutes); and
- decontaminate before disposal by chemical disinfection, steam sterilization, or incineration. In the authors work, small contaminations of laboratory work area or glassware where treated with a chemical disinfectant. Steam sterilization (minimum 121°C, 15 minutes) was used for bulk glassware or glass-fibre filters prior to washing.

Table 2Possible Infectious Substances in Swine Waste(adapted from Health Canada 2004)

Ascaris spp.
Balantidium coli
Burkholderia (Pseudomonas) pseudomallei
Campylobacter jejuni, C.coli, C. fetus
Citrobacter spp.
Clostridium botulinum
Clostridium difficile
Clostridium perfringens
Clostridium tetani
Cryptosporidium parvum
Diphtheroids
Edwardsiella tarda
Enterococcus faecalis, Enterococcus faecium
Escherichia coli, enterohemorrhagic, enteroinvasive, enteropathogenic, entertoxigenic
Fusobacterium spp.
Giardia lamblia
Hantavirus
Klebsiella spp.
Leptospira interrogans
Listeria monocytogenes
Salmonella choleraesuis
Salmonella spp.
Shigella spp.
Trichinella spp.
Vesicular stomatitis virus
Yersinia enterocolitica

2.5.2 Existing Guidelines for Water Reuse

The health guidelines provided by the World Health Organization provide recommendations primarily for the use of wastewater produced by domestic sewage and municipal wastewater plants (WHO 1989). The limit of <1000 FC/100 mL and <1 helminth egg/L for wastewater irrigation was published and is a common guideline used among international agencies. A tighter guideline of 200 FC per 100 mL was recommended for urban domestic irrigation of vegetables eaten raw but it was interesting to note that the Scientific Group concluded that a bacterial guideline is unnecessary when the only exposed population was farm workers, due to the lack of evidence. The guideline was challenged in 1992 by the stricter USEPA/USAID requirement, which called for no detectable FC/100 mL, BOD \leq 10 mg/L, turbidity \leq 2 NTU and a chlorine residual of 1 mg/L. The latter also called for rigorous engineering requirements for biological treatment, sand filtration, chemical disinfection, and back up features. Development of a risk management approach, based on mathematical and experimental data, indicated that the USEPA standard would not provide significantly greater protection and an estimated cost 4.5 times the WHO guideline (Shuval, et al 1997). Quality factors were documented surrounding the applicability of reclaimed water use (Crook 1991). Crook's considerations not only included public health protection, use requirements, irrigation effects and environmental considerations but the less science based considerations of aesthetics, public/user perception and political realities. Not surprisingly this is reflected in the widely variable requirements for reclaimed water throughout the world.

There are issues in potable reuse that have overshadowed the technical possibilities of direct reuse. While most of these issues are related to human reuse of reclaimed wastewater, they may control the formal acceptance of reuse for livestock watering. The viability of augmenting drinking water supplies with reclaimed water was evaluated and provides insight into what measures would be necessary to gain official acceptance. "Direct use of reclaimed wastewater for human consumption, without the added protection provided by storage in the environment, is not currently a viable option for public supplies (National Research Council (U.S.). Committee to Evaluate the Viability of Augmenting Potable Water Supplies with Reclaimed Water. 1998). Many cities, towns, farms and rural residents already use raw water whose quality is impacted by upstream discharges of wastewater for indirect potable reuse. The National Research Council in the US states that more than two dozen major water utilities use river water that receive wastewater discharges accounting to more than 50% if the stream flow during low conditions. Their general conclusion was that "indirect potable reuse is a viable application of reclaimed water, but only where there is a careful, thorough, projectspecific assessment that includes contaminant monitoring, health and safety testing, and

system reliability evaluation". All water conservation and non-potable reuse options should be examined first. In regard to microbial contaminants in reuse systems, a specific reference was made to the new membrane water filtration systems, which can almost completely remove microbial pathogens. They felt that experience with them was not yet adequate and a strong chemical disinfectant, such as ozone or free chlorine should be used in these systems. The multibarrier approach to contamination risk is strongly advocated.

A review of water reuse and recycling, with reference to Canadian practice and potential was recently undertaken. "As compared to other countries worldwide, water reuse is currently practiced infrequently in Canada, with the focus of most of the water reuse effort within Canada on agricultural irrigation applications (Exall, et al 2004)". In Canada there are no national water reuse guidelines or criteria but well established standards and criteria do exist, for example, Australia and parts of United States (Marsalek, et al 2002). British Columbia and Alberta have developed regulatory guidance documents for water reuse. Water use in agricultural livestock facilities loosely comes under the blanket of potable reuse. Direct potable reuse of treated wastewater is rare and mainly studied at a feasibility level. On-site wastewater reclamation and reuse is practiced to some extent with the most common types of reuse being toilet flushing and agricultural or landscape irrigation. It was stated that membrane technology was well suited to decentralized water reuse in that they can produce a high quality effluent in a compact system and are tolerant to variations in solids concentration. Fouling behavior needs to be controlled by pretreatment and physical or chemical cleaning methods. Bioreactors in combination with membrane filters can convert foulants from soluble form into filterable biomass.

2.5.3 Bulk Treatment to Reduce Contamination Risk of Livestock Waste

Treatments with potential for inactivating viruses in pig slurries (dilute and concentrated) were reviewed to determine possible treatments for large scale swine waste decontamination (Turner and Burton 1997). Studies of natural inactivation of viruses in stored sludges showed the process to be slow, probably in the order of months. Seventeen methods including physical, chemical and biological treatments were assessed with one criteria of suitability being a minimum 10⁴ fold reduction in infectious units. A common

reason cited for reduced effectiveness of treatments is the protection of viruses by organic matter or by adsorption on to particulate matter. Viruses were often more resistant to inactivation than bacteria so longer treatment durations and/or doses are expected. The most suitable treatments were the use of heat (60°C, 30 min.) or the application of sodium or calcium hydroxides, or formalin at appropriate concentrations. Plug flow designs may be more appropriate to improve contact time and reduce recontamination of treated wastes. Membrane filtration was not specifically mentioned but would fall into the application of treating dilute wastes. Reducing suspended solids and turbidity is key to the effectiveness of most physical and chemical treatments assessed. The exception was heat treatment where the higher solids content was felt to improve the retention of heat thereby aiding virus inactivation.

2.5.4 Membrane Integrity

Microfiltration of wastewater provides an effective barrier to most microbial pathogens that could exist in animal wastes or wastewater. Ultrafiltration may be required to exclude some enteric viruses. Unlike chemicals like chlorine, there is not an ongoing resistance to the potential reintroduction of them in the system. To be assured of dependable membrane system results, a means of monitoring its integrity must be built into its operating schedule. A variety of direct and indirect methods to insure membrane integrity have been developed but each has its own advantages and disadvantages. The low-pressure, outside-in membrane system is a relatively recent development and no single test available has immerged to fulfill all integrity monitoring requirements (Farahbakhsh, et al 2003). Currently the standard test for detecting minor breaches of membrane integrity is the pressure hold (decay) test whereas particle counting or turbidity monitoring have been used to meet regulatory requirements or detect more major breaches.

3. MATERIALS, EQUIPMENT AND PROCEDURES

3.1 Collecting and Preparing Raw Wastes

Swine wastes used in this membrane treatment were obtained from the Swine Research and Technology Centre located on the University of Alberta's Research Station in Edmonton. This new facility completed in 2002 houses a sow herd equivalent of 220 with significant biosecurity measures built into the design and practiced. Swine wastes are stored as liquid manure typically 95% moisture content. Wastes build in up in the various animal production zones and are drained under manual control to a collection tank and lift station located immediately adjacent to the barn. From here wastes are pumped as needed to composting facility or loading system for field application. In a farrow to finish swine production facility the mature animal herd will produce the greatest amount of waste with the highest moisture content. By coordinating collection times with the barn manager the collection of fresh and representative swine wastes from this mature population was targeted.

Samples were transferred with a portable submersible pump to two 220 litre PVC barrels outfitted with two to three sampling ports on vertical axis. These were filled and transported to a nearby research building (F52) where they could remain at 15 to 20°C undisturbed for primary natural settling. These were sealed and only the headspace was aerated at a low level to avoid accumulations of hazardous gases. Plate 1 shows this procedure. Within 24 to 48 hours, approximately 80 L of SWSN was drawn from each barrel below the liquid surface in the top 250 mm into 20 L PVC pails. Pails were sealed and identified as to their collection height and time, then transferred to a cold room maintained at 4 °C in the Environmental Engineering building near where the membrane filtration system was located. Plate 2 shows the vessel used for initial settlement and separation of SWSN.



Plate 1 Collection of Swine Wastes



Plate 2 Settling of Swine Wastes Prior to Collection of SWSN

3.2 Bench top system

The hollow immersed membrane studied was a low pressure, outside-in ZW500d© membrane manufactured by Zenon operating in a dead end microfiltration mode. This membrane module has undergone two Environmental Engineering Program research projects to evaluate the feasibility of using it in a membrane bioreactor (Heise 2002) and a recent study of integrity testing and monitoring (Farahbakhsh, Adham and Smith 2003). The polymeric fibers used in this module belonged to a supported, non ionic, hydrophilic microfiltration membrane, whose membrane structure was asymmetrical with inside diameter and nominal pore size of 2 mm and 0.04 μ m, respectively. See Plate 3. A suggested schematic for a bench top test arrangement was provided with the membrane module from the manufacturer, Zenon Environmental, and design altered to fit the particular application requirements. The ZW-1 membrane module can be used for preliminary investigation into the performance of commercial ZeeWeed[®] 500 modules. A schematic is shown in Figure 2 with picture plates following. Three main features were added to this system and are identified below. Table B1 in Appendix B contains a listing of the equipment and supplies required to build the operational components of the system. Where specialized calibration equipment was required it is shown beside the device it was used to calibrate.



Figure 2 ZW-1 Bench Test System



Plate 3 ZW-1 Module (adapted from Zenon Environmental Enterprises Ltd. 2001)

3.2.1 Data Acquisition and Control (DAQC) System

A pressure transmitter sent voltages corresponding to pressures measured immediately downstream of the membrane module on the permeate side. This signal was picked up along with two additional voltages from temperature thermistors located in the process and permeate tank and transferred to an analog processor and then to digital input/output board installed in the computer. The computer also communicates to the peristaltic pump through the COM1 port via RS-232 cable and in real time gathers operational data and controls the membrane filtration process (pump rpm and direction) to stay within design parameters. This monitoring and control requirement was facilitated through a DAQC program called Softwire 3.1 which allows the use of Visual Basic programming to perform iterative logic steps to control the system. Figure 3 provides a basic schematic of the DAQC system and a summary of the key operating parameters and control algorithm.
Appendix B contains greater detail including Figure B1 custom control screen, Figure B2 design wire diagrams (4 pages) and Figure B3 VB 6.0 program coding. Plate 4 shows the components of the DAQC system and an overall picture of the system layout.



Plate 4 Computer, Sensor Connection and Overall Layout



Figure 3 DAQC System, Inputs and Control Algorithm

3.2.1.1 Calibration of System Sensors

The primary sensor measurement controlling the membrane filtration was pressure. This measurement was made on the permeate side of the membrane as indicated in Figure 2. A compound pressure transmitter (EW-68848-26) with an operating range of -101.4 to 207 $kPa \pm 1\%$ (- 14.7 to 30 psig) passed a 1 to 5 volt signal to the computer. The voltage read at the computer was calibrated against a known pressure exerted on the transmitter. A Druck DPI 1603 pressure calibrator was used to calibrate this relationship from -68.9 to 68.9 kPa (-10 to 10 psig). The equation describing this relationship was input into the DAQC system to convert voltages read to pressures. Figure A1 in Appendix A provides the calibration results to produce this pressure vs. voltage relationship. A pressure snubber with a sintered metal diaphragm was used to protect the pressure transmitter from shocks and regularly cleaned with a 10% HNO₃ solution to avoid incrustation. Figure A2 in Appendix A provides the calibration results for the backup pressure gauge used in system runs not requiring continuous real-time data collection.

Temperature measurements were made with immersed thermistors in the process and permeate tank and the calibration and system integration is described in 4.3.5. Calibration curves are provided in Figure A3 and Figure A4 in Appendix A.

3.2.2 Cross Contamination Control and Sample Collection

All feedwater, process water, and permeate flows were contained within tubing which could be replaced between runs or disinfected with a 200 mg/L NaOCl solution and rinsed with demineralized water (DI). Sample ports were located so that in-line samples could be drawn from the tubing during the permeate production phase of the filtration cycle. A backflush vessel was located upstream of permeate tank so that filtered water would not come in contact with open air and potentially contaminate sample.

After each run with dilute SWSN the tanks, floats, and tubing were emptied and disinfected. The membrane module was removed, rinsed with DI and immersed in approximately 200 mg/L NaOCl solution between runs.

3.2.3 Odour Control

A tank within a tank concept was used to contain potential spills of this SWSN with its strong biohazard potential and facilitate odour control. The process tank was under slight positive pressure due to the aeration required for the filtration module. The flexible dryer venting tube passively connected to the labs fume hood was sufficient to mitigate odours once lids were replaced during system runs.

3.2.4 Aeration of Membrane Module

The ZW-1 module requires aeration to provide air bubble scouring of the membrane straws and reduce fouling behavior. In larger modules this aeration also agitates the membrane straws to provide additional physical scouring. The maximum airflow rate recommended for the module was 1.7 m^3 /h. Air supply was provided from two Type T tanks containing normal air through a dual regulator and flow rate measured through an airflow rotameter. This rotameter was checked for accuracy with a wet test meter. Plate 5 and Plate 6 illustrate the layout of tankage, installation of membrane and odour control venting.



Plate 5 Tankage and Membrane Module Installation



Plate 6 Odour Control Venting From Tanks to Fume Hood

3.3 Operating Procedures

Two types of monitoring were carried out during filtration runs – performance data and treatment effectiveness.

3.3.1 Performance Data Collection

After initial flow rates were set, performance data was collected and recorded to Excel files on a real time basis every five seconds. Based on the pressure measured the system would adjust so as not to exceed maximum pressure specifications. To counteract the effects of fouling a backflush cycle was set (flow rate, duration) and manually adjusted in the early stages to what appeared minimum levels. Sensors inputs were scanned at 1000 Hz for 200 counts per channel and assessed every 5 seconds by the control program for pumping rate, initiation of backflush cycles or system shutdowns if operating out of specified limits. Systems runs were performed in batch mode and continued until either 10 L of permeate were produced or it was demonstrated that the system could not recover from membrane fouling. To approach the best run with this raw water a baseline run with the two modules was performed with DI then additional runs at 9%, 20% and 30% SWSN by volume. The pump speed and direction was controlled with command strings

sent to the pump from the computer via the COM1 serial port. The strings were customized to the ASCII format provided in the operator's manual of the 7550-20 Masterflex L/S pump. The author provided parameter values and algorithm for the operating strategy.

A nomenclature for all Excel data files produced during runs was used and is defined as follows:

"ZW₁ D_{20} F₅₀" means #1 ZW-1 module used at a dilution of 20% SWSN and an initial flowrate of 50 mL/min set.

3.3.2 Sample Collection and Testing

In most cases, a running start was performed in which the system was started on DI and then well mixed SWSN was added to the feedwater and process tank at approximately 5 minutes. Minor spills were absorbed with paper towel, which was disinfected with bleach solution prior to disposal. Remaining effluents or sample waste were disinfected and sent to drain or repailed and transferred back to waste handling system of swine facility.

Turbidity, conductivity, pH and temperature were sampled and measured at regular intervals of permeate production to monitor performance of the filtration. TSS, TDS and COD were also sampled for analysis at the same interval during complete runs. Process water could be drawn from the vicinity of the membrane module with a separate peristaltic pump. Permeate grab samples were collected from an in-line sampling port or composite samples from the permeate collection tank. Collection methodology will be identified in the analysis description.

3.4 Analysis of Samples

All wastewater quality parameters were analyzed and samples stored according to <u>Standard Methods for the Examination of Water and Wastewater</u> (APHA 1999). A summary table of the methods used is shown in Table 3. General descriptions, apparatus required, and apparatus are provided for each analysis used.

Analysis	Standard Method #	Abbreviation	Units
Total Suspended Solids	2540 D	TSS	mg/L
Total Dissolved Solids	2540 C	TDS	mg/L
Chemical Oxidation Demand	5220 D	COD	mg O ₂ /L
Turbidity	2130 B	Turb.	NTU
Temperature	2550	Temp.	°C
Conductivity	2510B	Cond.	mScm ⁻¹ , µScm ⁻¹
pH	4500	pН	
Total Kjedahl Nitrogen,	4500-N _{org} B	TKN, NH ₃	mg/L
Ammonia			
Total Phosphorus	4500-P	ТР	mg/L
Total Organic Carbon	5310B	TOC	mg/L
Total Sulphide	4500-S ²⁻	S ²⁻	mg/L
5-Day Biochemical Oxidation	5210B	BOD ₅	mg O ₂ /L
Demand	4500-O C		
Fecal Coliform	9222 D	FC	CFU/100mL
Total Metals	3030 E, 3110	n/a	mg/L

Table 3Summary of Analytical Methods Used

3.4.1 Turbidity

A well mixed process water and permeate sample were regularly measured whole for turbidity to help identify possible deviations of filter performance during continuous system operation. Turbidity in the nephelometric method used is a comparison of the light scattered under defined conditions to the light scattered of a reference standard under the same conditions. The higher the intensity of light scattered the higher the turbidity (APHA 1999). This scattering of light is caused by the optical interference of suspended and colloidal particles such as the dispersed organic matter or microscopic organisms present in dilute SWSN.

3.4.1.1 Apparatus

- Orbeco-Hellige Direct Reading Turbidity Meter
- 40 NTU Standard formazin

3.4.1.2 Procedure

A well-mixed 200 mL grab sample was collected in 250 mL beaker from process tank and in-line sampling port of permeate line (during permeate production) and measured within 20 minutes for turbidity, conductance, pH and temperature.

Turbidity meter was warmed up, zeroed and calibrated in 0 to 99.9 range with well dispersed 40 NTU standard. Two measurements of each undiluted sample taken were initially performed to verify that technique would produce repeatable measurements with wastewater used. Measurements for process water were taken in 0 to 999 range and permeate water in 0 to 9.99 range. Samples were close to room temperature so no condensation on sample vials was apparent. Gentle agitation was applied to the sample just prior to transfer to measurement vial to avoid effects of rapidly settling particles.

3.4.2 Conductivity

Conductance is the measure of an aqueous solution's ability to carry an electric current. This is predominantly affected by the concentration, mobility and valence of the ions present and the temperature of the aqueous solution (APHA 1999). This property is directly related to the degree of mineralization present and can be readily measured in many situations. Organic material, which is not fully dispersed, will reduce the conductivity measured in a solution. A consistent relationship can be established between conductance and TDS between 0.55 and 0.9. Conductivity is measured in mS/cm. (milli-Siemens per centimeter). The actual reading sensed by the meter is automatically corrected to its value at 25°C because of the temperature dependence of the conductivity measurement.

3.4.2.1 Apparatus

- Accumet AR20
- Conductivity probe: 4-Cell, glass body, Cell constant = 10.0 cm^{-1}

3.4.2.2 Procedure

A well mixed 200 mL grab sample was collected in 250 mL beaker from process tank and in-line sampling port of permeate line (during permeate production) and measured within 20 minutes for turbidity, conductance, pH and temperature.

The conductivity probe was calibrated to a 0.01 M KCl solution, which has a conductance of 1412 μ S/cm at 25°C. The probe is rinsed with DI and stored in the same between measurements.

3.4.3 Total Suspended Solids (TSS) and Total Dissolved Solids (TDS)

Gravimetric methods were used to determine TSS and TDS. A well mixed sample is filtered through pre-weighed ceramic filter holder (Gooch) containing a 1 μ m pore size glass fiber filter. It was then dried to a constant weight at 103°C to 105°C and reweighed. TSS is calculated as the difference in weight of the filter divided by the sample size. TDS is calculated from the difference in weight of an evaporation dish, after an accurate volume of filtrate has been dried at 103°C to 105°C.

3.4.3.1 Apparatus

- Gooch crucibles with Gooch crucible adapter: oversize design, 25 mL
- Filter: A/E Glass Fiber, 1 µm pore size, 33.8 mm diameter
- Evaporation dishes
- Vacuum flask, Pyrex, 500 mL
- Vacuum filtration apparatus
- Drying oven: maintained 103°C to 105° C
- Volumetric pipettes
- Desiccator: with Drierite desiccating medium, anhydrous calcium sulphate
- Analytical balance: Mettler AE 166

3.4.3.2 Procedure

A 125 mL grab sample was collected from process tank and in-line permeate sampling port (during permeate production) into clean 125 mL Nalgene sampling bottles. 10 mL of each bottle was transferred to volumetric flask for COD determination.

Glass fibre filters were seated into Gooch crucibles with three doses of approximately 10 mL reagent grade water while on the vacuum apparatus. They were then dried at 105°C for at least 1 hour then stored in dessicator until use (at least 4 hours). Prepared filters were weighed to the nearest 0.1 mg prior to sample filtration for the determination of TSS and production of filtrate for determination of TDS. From 10 to 50 mL of process or permeate sample, respectively, was transferred from nalgene sample bottles to filter assembly under vacuum using volumetric pipettes. The vacuum flask was disconnected and 10 or 20 mL of filtrate transferred volumetrically to a pre-weighed evaporation dish for the determination of TDS. The filtration system was reassembled and the filter with suspended solid was rinsed three times with reagent grade water. Vacuum flasks were rinsed three times with DI between subsequent samples. Evaporation dishes and filters were then transferred to oven for overnight drying at 103 to 105 °C. They were transferred to desiccators for cooling and stabilizing to constant weight for a minimum of 3 hours before being weighed. The dishes and crucibles were weighed to the nearest 0.1 mg on the analytical balance. Samples were filtered and analyzed in duplicate and the average taken as the final result.

3.4.4 Temperature

Various temperature measurements were made during the course of the experiment as temperature has been shown to be a significant factor in membrane flux. It was measured with custom built immersible thermistors as part of the systems data acquisition and control system. These were precalibrated against a high precision mercury type thermometer and Fluke thermocouple sensor. As well the conductivity probe has built in temperature compensation. The pH probe used has automatic temperature compensation (ATC) and pHs are reported at 25°C.

3.4.4.1 Apparatus

- NTC Thermistors KC005E-ND (Digikey)
- 5.00 kilo-ohm Resistors 800 344-4539 (Digikey)

3.4.4.2 Procedure

A well mixed 200 mL sample was collected in 250 mL beaker from process tank and inline sampling port of permeate line (during permeate production) and measured within 20 minutes for turbidity, conductance, pH and temperature. Temperatures were also recorded as part of process control.

Thermistors were wired into a balanced bridge circuit and calibrated in an ice water bath which was slowly heated to 35 °C and measurements taken every 5 °C. Calibration curves produced are provided in Figure A3 and Figure A4 of Appendix A. Equations from these relationships were used in the DAQC system for temperature conversion from voltages measured. The thermistors were immersed in process and permeate tanks and continuous measurements taken. All other temperature dependent measurements such as conductivity and pH were taken with temperature correcting probes.

3.4.5 pH

Practically every phase of water and wastewater treatment is aided by the measurement of pH including the pH dependent processes of acid-base neutralization, water softening, precipitation, coagulation, disinfection, and corrosion control (Sawyer, et al 1994). A potentiometric measurement using a standard hydrogen electrode and a reference electrode produces a plot of the electromotive force (emf) against the pH of different buffers. Subsequently sample pH is determined by measuring the emf produced.

3.4.5.1 Apparatus

- Accumet AR20
- Glass body combination probe (temperature compensating)

3.4.5.2 Procedure

A well mixed 200 mL sample was collected in 250 mL beaker from process tank and inline sampling port of permeate line (during permeate production) and measured within 20 minutes for turbidity, conductance, pH and temperature.

A three point calibration (pH = 4, 7, and 10) of the probe was carried out. pH measurements were automatically adjusted by the meter to 25°C. Hydrogen sulphide and ammonia are interferences in this method. Their presence as dissolved gases contribute to acidity in the wastewater and it is important to promptly measure the pH before changes occur. DI also has no buffering capacity to pH change so this can occur rapidly. Proteins, which will be present in swine manure, will release amine groups, which will combine with hydrogen ions to form ammonia and cause pH to rise initially.

3.4.6 Chemical Oxidation Demand (COD)

Chemical oxidation demand is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions. The quantity of oxidant consumed is expressed in terms of its oxygen equivalence. Because of its unique chemical properties, the dichromate ion $(Cr_2 O_7^{2^-})$ is the specified oxidant in the method used here (APHA 1999). The closed reflux method was used for culture tube preparation and colorimetric measurements made after.

3.4.6.1 Apparatus

- Spectrophotometer: Novaspec II
- Digester: HACH COD reactor
- Micro reflux tubes with seals: HACH, 10 mL
- Volumetric pipettes, 2 mL
- Graduated transfer pipette

3.4.6.2 Procedure

A 125 mL grab sample was collected from process tank and in-line permeate sampling port (during permeate production) into clean 125 mL Nalgene sampling bottles. 10 mL of

each bottle was transferred by pipette to clean 100 mL volumetric flask for dilution prior to COD determination.

A mixture of three solutions is required in each micro-reflux reaction tube when performing this procedure.

- 3.5 mL of digestion reagent (10.216 g K₂Cr₂O₇, 33 g HgSO₄, 167 mL concentrated H₂SO₄, diluted to 1000 mL with regent grade water)
- 2.0 mL COD reagent (9.715 g Ag_2SO_4 / L H_2SO_4)
- 2.0 mL diluted sample. (10 mL of sample was volumetrically diluted with reagent water to 100 mL to form a 10% solution)

Micro-reflux tubes for triplicates of samples and standards and a duplicate for a DI blank were capped and mixed three times and placed in COD reactor for 2 hours at 140°C. Tubes were mixed three more times and placed in tube rack in dark to cool for at least 1 hour. A spectrophotometer was warmed up during this cooling period and absorbance at 600 nm was then measured using the DI blank to zero the spectrophotometer. A six point standard curve was produced by digesting a potassium hydrogen phthalate (KHP) standard under the same conditions at concentrations of 50, 100, 200, 300, 400, and 800 mg/L KHP. See Figure D1, Appendix D. COD values for samples are determined by comparing absorbance at 600 nm to that of the standard curve and applying the theoretical factor of 1.176 mg $O_2/$ mg KHP.

Samples were analyzed in triplicate and the average taken as the final result. Samples that could not be analyzed within 24 hours were acidified to pH < 2 and stored at 4°C. These were subsequently analyzed within three days

3.4.7 Total Organic Carbon (TOC)

Measurement of TOC is of vital importance to the operation of water and waste treatment plants (APHA 1999). Organic carbon may serve as a nutrient source for biological growth on membranes. Disinfection byproducts can be formed when organic compounds

react with disinfectants to produce potentially toxic and carcinogenic compounds. Substantial quantities of organic carbon are present in livestock wastes.

To measure total carbon (TC) the high temperature (680° C) combustion method is used to convert all covalently bonded carbon to CO₂ where it can then be picked up in an oxygen gas flow and be read by a non-dispersive infared detector. The TIC fraction contains carbonate, bicarbonate, and dissolved CO₂ and can be measured separately from TC by acidifying the sample and running through the same detector. TOC is the difference between total carbon (TC) and total inorganic carbon (TIC).

3.4.7.1 Apparatus

• Dohrmann Carbon Analyzer (DC-80)

3.4.7.2 Procedure

A 125 mL sample was collected from process tank and in-line sampling port of permeate line into 125 mL acid washed and rinsed glass bottles and stored for later analysis as indicated in <u>Standard Methods</u> (APHA 1999).

The carbon analyzer required at least two hours of warm up prior to analysis and is operated according to the Dohrmann DC-80 Total Organic Carbon System Manual by an experienced technician to avoid problems. Thirty percent of the TC samples could be measured at full strength with the remainder needing 50% dilution. Samples were measured in reference to a 400 mg/L potassium phthalate (KHP) standard. All TIC samples could be run full strength and are measured in reference to a 400 mg/L Na₂CO₃ standard. A 40 μ L sample was injected where it was oxidized and the oxygen carrier gas delivered it to the infared detector. An electrical output was produced over a short time span and the unit calculated the area under the curve and adjusted the value to sample size to determine the mg/L of carbon. Samples were run in triplicate and the result was the average of the values.

3.4.8 5-Day Biochemical Oxygen Demand (BOD₅)

Whereas COD and TOC gives us a measure of the organic content of a water or wastewater, BOD analysis gives a measure of the potential impact of the water when

released into an aquatic environment. BOD is defined as the amount of oxygen consumed by bacteria when stabilizing decomposable organic matter under aerobic conditions (Sawyer, McCarty and Parkin 1994). Wastewater derived from swine wastes has large BOD values and a good measure of it requires important bacterial acclimatization and dilution steps. The 5-day BOD test has become a standard measure of pollution, as two thirds of the pollution strength of a sample will be exhibited within this period if suitable conditions are maintained. Since ammonia is a major constituent in swine waste the 5 day period is often chosen to limit interferences due to the oxidation of ammonia.

3.4.8.1 Apparatus

- Air incubator room ($20 \pm 1^{\circ}$ C)
- BOD Incubation bottles (300 mL) with flared ground glass stoppers
- Titration apparatus
- Magnetic stir plate
- YSI Model 50B Dissolved Oxygen (DO) meter
- Dilution water vessel (Nalgene, 40L) with air stone

3.4.8.2 Procedure

A 1 L composite sample was collected into clean 1 L Nalgene bottles (wide mouth) from process tank and permeate collection tank and prepared for dissolved oxygen analysis (Day 0) or incubation (Day 5) as indicated in <u>Standard Methods</u> (APHA 1999).

An acclimatized bacterial seed culture suitable for the SWSN was cultured over 6 weeks prior to use. Continuous aeration and addition of 1 g glucose every three days was maintained. SWSN was added periodically to maintain volume at approximately 600 mL.

Dilution water was prepared within 24 hours of test by aerating DI that had bacterial maintenance compounds at a rate of 1 mL/L added as per <u>Standard Methods</u> (APHA 1999) (phosphate buffer solution, magnesium sulphate solution, ferric chloride solution, calcium chloride solution). Sample analysis began during or immediately following system run. Dilutions of samples 10^{0} , 10^{-1} and 10^{-2} were prepared volumetrically.

Standard, DI blank, and DI blank with seed were tested in duplicate. Initially the DO was measured with a YSI DO electrode but this was abandoned for an Azide Modification of the Winkler method (4500-O C) due to variable results seen with the meter. Cleaned and rinsed incubation bottles all received seed (0.5 mL) except for dilution water blank to ensure ample bacterial culture was present. Sample triplicates and standard, blank and seed duplicates were prepared. Day 5 analysis incubation were transferred to darkened room at 20 ± 1 °C and Day 0 DO measurement completed as per <u>Standard Methods</u> (APHA 1999).

Averages were determined for duplicate and triplicate measurements and used as the result.

3.4.9 Total Kjeldahl Nitrogen (TKN), Ammonia (NH₃)

Nitrogen shows up in many forms, both inorganic and organic and is a key component of many of the life processes. The forms nitrogen resides in can provide us with an important measure of the potential results or bottlenecks that may be encountered in a process design. Nitrogen data has a long history as an indicator of sanitary quality in aquatic systems but more recently has been studied to determine nutritional balances, its oxidation in surface waters and control of biological treatment processes (Sawyer, McCarty and Parkin 1994). This will become especially important if the work on membrane treatment completed continues on from essentially direct filtration to a membrane bioreactor (MBR) mode. Nitrogen and its forms can be determined by a variety of methods and the volumetric method is a modification to the Standard Methods (4500-NH₃) that exists (APHA 1999). Basically TKN is determined by digesting the sample at high temperature and acidity to ensure all N is in the ammonium form. This can be converted into ammonia by distilling and raising the pH to alkali conditions. A receiver solution converts it back to the ammonium ion form and a titration is done with a strong acid to determine how much N is present. Inorganic nitrogen is determined in similar fashion but without the digestion or pH increase. Organic nitrogen can be calculated as the difference between these two.

3.4.9.1 Apparatus

- Tecator Kjeldahl 2020 for TKN digestion
- Tecator 1026 for TKN and NH₃ distillation
- Mettler Toledo DL50 Autotitrator
- 250 mL digestion tubes

3.4.9.2 Procedure

A 1 L composite sample was collected into clean 1 L Nalgene bottles (small mouth) from process tank and permeate collection tank and prepared for storage and later analysis as indicated in <u>Standard Methods</u> (APHA 1999). Samples were adjusted to pH<2 using concentrated sulfuric acid and stored at 4°C until analysis.

Initially an appropriate dilution had to be determined to put the measured values within an acceptable range of the method used. The SWSN used as process water was originally diluted to between 10% and 30% of its original strength so a one step dilution to 10% of sample strength was all that was required. From this 5 and 10 mL were the best sample sizes.

A digestion step is required for the TKN test and the author used the Tecator Kjeldahl 2020 digestion apparatus, which holds 250 mL digestion tubes. To reach the temperature and acidity required each tube received a boiling rod, two Kjeltabs (Anachemia # CT-37), 3.5 K₂SO₄ plus 0.4 gm CuSO₄ each), 100 mL sample plus reagent water together. 12 mL of concentrated sulfuric acid was added to each of 20 tubes under the fume hood and placed in holder. Then this block of 10 duplicates (20) containing blanks, standards, samples and/or placeholders are transferred to the preheated digestion block at 420°C (as outlined in the Tecator block digestion procedure) where vacuum apparatus is attached. Two hours is required for the digestion followed by a minimum of 1 hour cooling outside of the unit to prepare them for distillation.

The Tecator 1026 was required for distillation for both the digested TKN samples and NH₃ tubes using the procedure provided in the operator manual. In the latter the tubes

only contained 50 mL of sample and reagent water together. The Tecator 1026 automatically adds 75 mL of DI (containing indicator phenolphthalein) and 100 mL of 40% NaOH followed by steam injection. Approximately 100 mL of the condensate was then automatically transferred to a receiver solution of 25 mL of 4% Boric acid in a 250 mL beaker. The solution with N now in the ammonium ion form is then ready for titration.

The Mettler Toledo DL50 autotitrator was used for endpoint titration according to procedure provided in operating manual using 0.005 N HCl which has been standardized against 0.005 N Na₂CO₃. The pH probe with the autotitrator was calibrated against three commercial pH buffers (4, 7, and 10). The endpoint to be reached was made by determining the stable pH reading of a solution of 25 mL of the 4% Boric acid diluted to the same volume produced in the distillation step (about 125 mL). The unit was then programmed to titrate all blanks standards and samples to this endpoint value and runs completed. Each run produced a printout of the endpoint titration.

The distillation of the ammonia samples was the same except that the addition of 100 mL of 40% NaOH was not required to adjust the pH for the acidity produced in the TKN digestion step.

All samples, standards and blanks were performed in duplicate and average used as the result unless system error was observed in the operation of the digestion, distillation or titration units. This was infrequent.

3.4.10 Total Phosphorus (TP)

Phosphorus occurs in natural waters and wastewaters almost solely as phosphates. Organic phosphates are formed mainly by biological processes and found in body wastes and may also be formed from orthophosphates in biological processes. Although phosphorus is essential for growth and can be the limiting nutrient in a water body their release to that water may stimulate the growth of photosynthetic aquatic micro and macro organisms in nuisance quantities (APHA 1999). Phosphorus analysis was comprised of two main steps; the conversion of the phosphorus form of interest to dissolved ortho-

phosphate by one of many digestion methods possible and the colorimetric determination of it. The persulphate digestion and the vanaomolybdophosphoric acid colorimetric method were used. Since sulphide can be an interference in this colorimetric method a spiked sample was used to determine its extent.

3.4.10.1 Apparatus

- Ultrospec 3000 UV/Visible Spectrophotometer (Phamacia Biotech)
- Accumet AR 20 pH meter
- Autoclave Oven: M/C 3322 and M/C 3233 Gravity Laboratory Sterilizer

3.4.10.2 Procedure

A 500 mL composite sample was collected into clean 500 mL Nalgene bottles (wide mouth) from process tank and permeate collection tank and prepared for storage and later analysis as indicated in <u>Standard Methods</u> (APHA 1999).

Initially an appropriate dilution had to be determined to put the measured values within an acceptable range of the method used. The SWSN used as process water was originally diluted to 10% to 30% of its original strength. A 50 mL aliquot is required in the digestion phase and it was determined that 8 mL of sample (plus 42 mL of reagent water) was suitable for this method. This produced a second dilution equivalent to 16% of the original sample obtained from the process and permeate tank.

An ortho-phosphate standard was prepared with KH_2PO_4 to produce a solution concentration equivalent to 50 mg/L PO_4^{-3} -P. A 7 point standard curve comprising 0, 2.5, 5.0, 7.5, 10.0, 15.0, 20.0 mg/L PO_4^{-3} -P was produced using the same digestion and colorimetric procedure. See Figure D2, Appendix D.

In the persulphate digestion step samples, standards and blanks were prepared together and autoclaved in foil capped Wheatmen bottles (125 mL), which were cleaned in 10% nitric acid and rinsed beforehand. Autoclaving required 30 minutes exposure time to produce a final volume of about 10 mL. After cooling phenolphthalein indicator was

added (1 drop) and neutralized to faint pink with 1N or 6N NaOH before proceeding to colorimetric analysis.

Contents of samples, standards and blanks were individually transferred to 100 mL acid washed volumetric flasks with 20 mL of vandate-molybdate reagent and diluted with reagent water to 100 mL. After at least 10 minutes color development they were ready for colorimetric measurement on Ultrospec 3000, which was pre-warmed for 1 hour and set at 470 nm. All measurements were performed in triplicate and average of values obtained used as the result.

Each sample, standard or blank was analyzed in duplicate. Spiked samples with 5 mL of standard solution added were also analyzed to determine possible interference described in <u>Standard Methods</u> (APHA 1999), especially sulphide.

3.4.11 Total Sulphide

Bacterial action can break down sulphates present in livestock wastes to produce odour causing and potentially toxic levels of hydrogen sulphide. Sulphates can come from sulphur rich feed proteins containing amino acids (i.e. methionine, cysteine) or water supplies with elevated sulphates. The sampling procedure and analysis for hydrogen sulphide was designed to minimize the loss of this compound due to volatilization or reaction with dissolved or atmospheric oxygen. A quantitative iodometric method was used which uses iodine to oxidize sulfide in an acid solution. This method is also used to standardize the other methods (APHA 1999). A sulphide standard (1.00 mg S^{2-/} mL) was produced as per <u>Standard Methods</u> (APHA 1999) and used within 1 week of preparation as indicated.

3.4.11.1 Apparatus

- Titration burette
- Stirring plate

3.4.11.2 Procedure

Grab samples for sulfide analysis were collected from process tank and in-line permeate tubes with a minimum of aeration or headspace into acid washed and rinsed Wheatman bottles (125 mL) with parafilm covers plus caps. 4 drops of Zinc acetate/100 mL was added to sample and adjustment to pH>9. Samples were stored at 4°C until analysis. Degassed water was prepared and used in all dilutions and rinsing required.

The supernatant above the ZnS precipitate was decanted and the remaining floc released below the surface of 20 mL of 0.0250 N Iodine solution. Samples were acidified with 6N HCl and titrated with 0.0250 N Na₂S₂O₃ using a few drops starch as endpoint was approached.

No analysis replicates were prepared as all of sample volume was used undiluted to obtain sufficient sulfide to measure by titration

3.4.12 Fecal Coliforms (FC)

The fecal coliform test has been a standard for many years as an indicator of risk posed to human by contact or drinking of polluted waters. Fecal coliforms are a subgroup of total coliforms and are present in the gut and faeces of warm-blooded animals. These generally include organisms capable of producing gas from lactose in a suitable culture medium at $44.5 \pm 0.5^{\circ}$ C (APHA 1999). FC shares many of the same procedures as the total coliform test but is more selective to estimate the risk posed within warm blooded animals. Sterile procedures and identifying appropriate dilutions are key to this procedure.

3.4.12.1 Apparatus

- Membrane filter: GN-6 grid, pore size 0.45 μm, 47 mm dia.
- Vacuum filters
- M-FC culture medium
- Pre-sterilized plastic petri dishes, 60 x 15 mm
- Bunsen burner

• Incubator: $44.5 \pm 0.2^{\circ}C$

3.4.12.2 Procedure

A 500 mL composite sample was collected into clean 500 mL Nalgene bottles (wide mouth) from process tank and permeate collection tank. Analysis was begun during or immediately following system runs. Transfers were made to peptone dilution bottles with sterile pipettes.

Optimal sample size was chosen from experimentation to yield 20 to 60 fecal coliform colonies per membrane. Samples from appropriate dilutions were transferred with sterile disposable pipettes to bottles containing 30 mL peptone solution, which had been autoclaved, cooled and capped prior to analysis. The complete contents of each bottle was filtered though sterile membrane filter (GN-6 grid, pore size 0.45 μ m, 47 mm dia) under vacuum. This gridded filter paper was transferred to petri dishes containing sterile agar (M-FC, Risolic acid), capped and incubated at 44.5 ± 0.2 °C for 24 hours prior to enumeration of colony forming units (CFU).

Samples were completed in triplicate and result taken as average of three unless culture exhibited outside contamination. Peptone dilution water and filter checks were run in duplicate before and after filter runs to check for contamination.

3.4.13 Total Metals

Metals can have a variety of potential impacts, beneficial, benign or toxic, and an analysis to determine the potential concentration is warranted. Their impact can depend on how they are presented to the environment as exemplified by some metals toxicity to fish in natural waters being acidified. Total metals are a measure of all extractable metals including inorganically and organically bound species. For this reason a rigorous digestion step is necessary to extract metals for complete quantification in the method used.

3.4.13.1 Procedure

A 125 mL sample was collected from process tank and in-line sampling port of permeate line into 125 mL acid washed and rinsed glass bottles, acidified with HNO₃ to pH<2, and stored at 4° C for later analysis.

Representative aliquot of well mixed samples and a DI control were acid digested according to Method 3030 E (APHA 1999) on site. A mixture of nitric acid and sample was transferred to a 150 mL Griffen beaker, covered with a ribbed watch glass and refluxed repeatedly with portions of HNO₃ until the digestate was light in color or stabilized. This continues until a low volume (3 mL) was left. Once cooled, 1:1 HCl was added, covered and refluxed for a short period (15 minutes) to dissolve any precipitate. The sample volume was adjusted to 100 mL with reagent water and 10% final acid concentration. Samples were transferred to 1% HNO₃ treated Nalgene bottles (125 mL) and taken to private lab for total metals analysis by atomic (ICPM) spectroscopy.

3.5 Statistical Methods

95% confidence intervals were determined for flux measurements of D₀ runs.

Descriptive statistics were determined for cycle times produced during D_{20} run and used to eliminate outliers not explained by system run observations.

Table D3, Appendix D provides descriptive statistics for reductions calculated for TSS, TDS, COD, turbidity and conductivity that were taken at regular intervals during the D_{20} runs.

Relative standard deviations were determined for analysis, and reported with data for TKN, NH₃, and TP.

4. **RESULTS**

As a representative of a hollow fibre immersed membranes, a microfiltration membrane manufactured by Zenon Environmental, was evaluated for potential operation in the organically rich environment of swine wastewater. The compact ZW-1 membrane module, contains approximately 0.047 m² of ZeeWeed[®] 500d membrane and configured in a 175 mm long X 58 mm diameter (Plate 3). It was immersed into 66 L of diluted SWSN. The process tank liquid elevation was maintained at a constant level of 425 mm by transfer from the feed water tank containing equally diluted SWSN. This transfer was synchronized with permeate flow out of the process tank. This semi-batch mode reduced the change in effluent quality parameters and as evidence COD concentration increased only slowly over the operating period. Although the manufacturer intended the module to operate at a fixed flux rate the author opted for a pressure controlled flux rate. This was done with a customized data acquisition and control system (DAQC) so that flux decay and cycle times could be examined. Pressure sensor calibrations, control routines or performance charts provided from system runs used pressure values in psig units. Calculated pressure values are shown in SI units (kPa) for summary or discussion purposes.

Two ZW-1 modules were obtained from the manufacturer in sealed packages with glycerin added to stop the membrane from drying out. Preparations, as indicated in the operating manual, included pressure testing for air leaks and overnight soaking in 200 mg/L NaOCl solution. Visual examination of the modules did not indicate differences in the virgin ZeeWeed[®] membrane. During aeration of the new vertically mounted membrane modules, the air bubbles were observed to exit the membrane sheath near the top and could affect overall membrane scouring by aeration.

4.1 Baseline Measurements in Distilled Water (D₀)

4.1.1 Change in Normalized Specific Flux

Both ZW-1 membrane modules were operated in distilled water without the addition of SWSN (D_0) at the beginning and end of runs with diluted SWSN. Maximum pumping capacity of the peristaltic pump used was reached before the maximum operating pressure 48.3 kPa (7.0 psig) or the 34.5 kPa (5.0 psig) recommended for baseline runs on the module was reached. Alternatively, each D_0 was run until a stabilized vacuum pressure was obtained (5 to 10 minutes) at four flow rates (100, 150, 200 and 233 (or 238) mL/min). Two sets of post D_0 runs (After 1 and After 2) were made (73 days apart) after the runs diluted with SWSN to evaluate fouling and recovery from fouling, The temperature, conductivity and pH of DI was similar in all D_0 runs and is tabulated in Table D1, Appendix D.

Temperature affects the viscosity of water and potentially the membrane flux attainable. All D_0 flux values were converted to a J_{20} unit expressed in L h⁻¹m⁻² kPa⁻¹ normalized at 20°C and presented in Figure 4. Table C1, Appendix C provides descriptive statistics with 95% confidence interval used for error bars.



Figure 4 Normalized Specific Flux of D₀ Runs

4.2 Performance of Membranes in Diluted SWSN

4.2.1 Operating Settings

The DAQC system allowed for any combination of initial flow rate, backflush rate and duration, and control routine settings to be evaluated. In the initial runs with 9% SWSN (D₉) with the ZW₁ module it was determined that an initial flow rate of 50 mL/min could be used. It was also determined that the backflush rate could be up to 50% greater than the initial flow rate without causing a stoppage. A backflush of 70 mL/min for 50 seconds was chosen. The backflush volume used is automatically subtracted off the total volume tracked. Manual records of permeate volumes produced and sample volumes removed from the system verified this was working. The same operating parameters were also used for 30% and 20% SWSN (D₃₀ and D₂₀ respectively) on both modules.

4.2.2 Performance Results in D₉ and D₃₀ Runs

After 103 minutes of run time in the D₉ run of ZW_1 , a flow rate of 31 mL/min was maintained with the ZW_1 module without backflush during the complete 285 minute operating period required to produce 10 L of permeate. In this case 9% SWSN

corresponded to an average COD of 1236 mg/L. By the end of the run, flux had reduced to $1.12 \text{ L h}^{-1} \text{ m}^{-2} \text{ kPa}^{-1}$ for the ZW₁ module.

After 145 minutes in the D₉ run with the ZW₂ module, a flow rate of 45 mL/min was maintained without backflush during the complete 213 minute operating period required to produce 10 L of permeate. In this case 9% SWSN corresponded to an average COD of 1368 mg/L. By the end of the run, flux had reduced to 1.25 L h⁻¹ m⁻² kPa⁻¹ for the ZW₂ module.

Figure C2 and Figure C3 in Appendix C show the operating runs from these lower concentration effluent treatments.

A 30% SWSN run (D_{30}) was attempted following the positive D_9 results. Three runs were attempted with each module to produce the 10 L of permeate targeted for runs. Only 1 of 3 runs for each module produced reportable results.

 ZW_1 was only able to produce 6.4 L of permeate after 383.5 minutes operation. See Figure C7 Appendix C. During the first 262 minutes, cycle times reduced from 175 to 140 seconds. The effects of reduced membrane aeration (1.7 to 1.1 m³/h) could be seen at 260 minutes. Partial recovery could be seen at 380 minutes when aeration was restored to previous level. More variation in cycle time could be seen and was another reason a reduced concentration on SWSN was used in the final runs. In this case 30% SWSN (D₃₀) corresponded to an average COD of 4854 mg O₂/L

 ZW_2 produced longer cycle times but still decreased from 260 to 190 seconds in 130 minutes or 3.8 L of permeate production. At this point the run was halted by the DAQC system to protect the membrane from over range suction pressures. See Figure C7, Appendix C. The trend in cycle times for ZW_1 and ZW_2 modules over the operating period are shown in Figure C8 and Figure C9 in Appendix C.

4.3 Best Run Performance (D₂₀)

4.3.1 Cycle Time and Duration of Backflush (D₂₀)

The DAQC system allowed the membrane to find its own equilibrium for cycle times between backflushes. A minimum sustainable backflush duration, intensity, and frequency which would provide the quickest production of 10 L of permeate were chosen as the target in this system. Three minutes or 180 seconds was arbitrarily targeted as the minimum cycle time for operation. A cycle time less than this reduces the net permeate production and could be hard on the pump with the frequency of direction changes. With a backflush duration set at 50 seconds and flow of 70 mL/min, this resulted in about 20% of the operation time being consumed in backflush. The length of cycle times (not including the time for backflush) is shown for the ZW₁ and ZW₂ modules in Figure 5 and Figure 6, respectively. The change in air tanks at 280 minutes as shown in Figure 5 caused an increase in the cycle time and will be discussed later.



Figure 5 Cycle Time for D_{20} Run of ZW_1



Figure 6 Cycle Time for D₂₀ Run of ZW₂

4.3.2 Normalized Specific Flux (D₂₀)

Instantaneous flux was always changing in the D_{20} runs unlike the equilibrium that was sustained in D_9 runs. An effective flux can be estimated taking the total time to produce 10 L of permeate. The operating run for ZW₁ shown in Figure C4, Appendix C and ZW₂ shown in Figure C5, Appendix C show operating times of 307 and 389 minutes, respectively. This produces an effective yield of 32.6 and 25.6 mL/min for ZW₁ and ZW₂, respectively. When adjusted for temperature and expressed in normalized J₂₀ values the following results are seen for the D₂₀ runs.

D ₂₀	Flow	Pressure	Temperature	J ₂₀
Units >	mL/min	kPa (psig)	°C	$L h^{-1}m^{-2} kPa^{-1}$
ZW_1	32.6	28.7 (4.16)	20.7	1.42
ZW_2	25.6	25.6 (3.71)	19.0	1.30

Note: Average pressures and temperatures over the run were used to normalize flux.

4.3.3 Fouling Characteristics

A characteristic pressure curve was observed during the cycles generated as illustrated in a segment out of the D_{20} run of ZW₁. See Figure 7. At the completion of the backflush, a quick drop in pressure occurs as the pressure from the previous backflush is releaved. This decline slows as the pump begins to exert a pressure differential on the membrane and permeate is produced. Typically a plateau was seen around -34.5 kPa (-5 psig) was seen before the pressure began to drop initiating another backflush cycle.



Figure 7 Representative Pressure and Flow During D₂₀ Cycle.

4.3.4 Aeration of Membranes

Early on in the test procedure it was confirmed that aeration was required to reduce the fouling behavior of the membrane in concentrated effluents. Aeration was kept at a constant rate of 1.7 m^3 /h for all recorded runs except for two instances where unplanned events occurred. Figure C8, Appendix C shows the effect of reduced aeration and Figure 5 is suspected to show the effect of an increase in airflow.

4.3.5 Temperature Monitoring

Temperature data from the process and permeate tanks was provided from the real time collection in D_{20} and D_{30} runs as well as those gathered as part of grab sample water quality measurements. Temp 1 were taken in the process tank and Temp 2 from the permeate tank. These values were used in the normalizing of baseline flux measurements of the modules in distilled water. A sudden drop in temperature results when the SWSN cooled to 4°C for storage is added to the distilled water. Temperature did not change significantly during the runs although a small downward trend likely caused by the aeration of the module can be seen.

4.4 Treatment Performance

Water quality measurements were made in all runs with the goal of determining what reductions in target parameters could be made when stable operating settings were determined for a maximum concentration of SWSN. The results reported here are those from the 20% SWSN concentration (D_{20}). Results from D_9 and D_{30} runs are summarized in tabular form in Table D2, Appendix D. Samples labeled PT refer to untreated water from process tank. IL refers to grab or composite samples taken from permeate. COD, TSS, TDS, pH, conductivity, and turbidity were measured periodically over the complete run to monitor membrane for potential failure or significant change in treatment levels. TOC, BOD₅, FC, TP, TKN, NH₃ and sulphides were measured from a sample taken halfway or 5 L through the run. The metal scan of a predigested sample from PT and IL from the highest concentration run (D_{30}) using ZW₁ was partially reproduced in Table D10, Appendix D.

4.4.1 Physical and Aggregate Properties

4.4.1.1 Turbidity

As shown in Figure 8 and Figure 9 turbidity was the only physical property measured to change significantly during the D_{20} runs. Turbidity in the untreated process tank increased from 319 to 424 NTU and from 286 to 362 NTU for ZW_1 and ZW_2 , respectively. This coincides with a 10 L removal of permeate from the process tank which was replaced with 10 L of SWSN at the same starting dilution (D_{20}).



Figure 8 Turbidity, Conductivity, pH and Temperature for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₁



Figure 9 Turbidity, Conductivity, pH and Temperature for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₂

4.4.1.2 Conductivity

Specific conductivity was consistent over time for process and permeate samples for both modules (See Figure 8 and Figure 9). This parameter reflects the TDS content in water or wastewater and can be masked by organic matter content. Since no significant change occurred during treatment, an explanation could be that the dissolvable solids were either fully dispersed by the initial dilution in the process tank or the level of dispersion did not change appreciatively over the run.

4.4.1.3 Solids

Duplicates of TSS were consistent over the runs except for one sample taken halfway through the treatment by ZW_1 . (See Figure 10 and Figure 11). This low value was not reflected in any other parameters measured at that time. Inadequate mixing was suspected. Suspended solids were effectively eliminated in the permeate during

microfiltration by the ZW-1 modules. Other parameters associated with suspended solids show some reduction.

The ratio of TDS to COD was very consistent over each run showing an average of 0.33 for ZW_1 and 0.41/0.43 for ZW_2 process tank and permeate tank samples respectively. This calculation is tabulated in Table D4 in Appendix D.



Figure 10 Total Suspended and Total Dissolved Solids for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₁



Figure 11 Total Suspended and Total Dissolved Solids for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₂

4.4.2 Aggregate Organic Constituents

4.4.2.1 COD

Both COD values and percent reductions during the membrane treatment were consistent over the run time by both modules as illustrated in Figure 12. A 31.5 % reduction (C.I. $_{95\%}$ 3.30%) and 32.7 % (C.I. $_{95\%}$ 3.61%) reduction were seen with ZW₁ and ZW₂ modules, respectively.



Figure 12 COD for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₁ and ZW₂

4.4.2.2 Total Organic Carbon

The TC and TIC triplicate measurements of process and permeate grab samples showed little variance within replicate and were used to calculate TOC values in the D_{20} runs of both membrane modules. Table D5, Appendix D shows the raw data and calculated values for this analysis. TOC values in the process tank were very similar between the two module runs and a reduction of 31.8 and 36.2% was observed in the filtration process by ZW₁ and ZW₂, respectively. Original TOC values of the SWSN prior to dilution were 28% higher in the D_{30} run than the D_{20} runs and showed 19.5% reduction in TOC during filtration.

4.4.2.3 5-Day Biochemical Oxygen Demand

 BOD_5 for diluted SWSN was measured for composite samples taken from the process and permeate tank during the D_{20} runs with ZW_1 and ZW_2 modules. Inconsistent dissolved oxygen results were obtained in earlier runs so the titration method was used for the final D_{20} runs. Triplicate measurements taken varied a maximum of 9.4% from the
mean in a sample measured for the permeate of ZW_2 but otherwise was less than 5% off the mean. Table D6, Appendix D provides the raw and calculated data from the D_{20} runs. The BOD₅ measured for the diluted SWSN was 20% greater for the ZW_1 run than the ZW_2 run.



Figure 13 Sulphide, TP, FC, NH₃, TKN, TOC, and BOD₅ for Process (PT) and Permeate (IL) Over D₂₀ Run of ZW₁ and ZW₂

4.4.3 Inorganic Non-Metallic Constituents

4.4.3.1 Total Kjedahl Nitrogen, Ammonia

Four TKN replicates and two NH_3 replicates were used in the analysis of process and permeate samples during the D_{20} run of each module. Figure 13 shows a reduction in TKN of 23% and 19% for modules ZW_1 and ZW_2 , respectively. Only a 12% reduction was seen in NH_3 in module ZW_2 . Raw data is reported in Table D7 and Table D8, Appendix D. The relative standard deviations reported in <u>Standard Methods</u> for nicotinic acid in the highest sample (20 mg N/L) was 0.84% and 3.03% (APHA 1999). A 50 mg N /L standard run with analysis showed a deviation of 1 mg N/L. for TKN and 2 mg N/L for NH₃. Five samples were also spiked with standard masses to evaluate the potential interference of nitrate on ammonia released from organic N during the digestion. No significant deviations from expected were observed.

4.4.3.2 Total Phosphorus

Data is tabulated in Table D9, Appendix D for TP analysis. Average data from process and permeate tanks for both modules are shown and a maximum relative standard deviation of 7.0% determined for the process water sample for ZW₂. The RSD precision for the persulphate digestion plus vanadomolybdophosphoric acid method for a 10.230 mg/L orthophosphate sample is reported in <u>Standard Methods</u> at 6.5% (APHA 1999). Potential negative interference of sulphide on TP concentration was evaluated using four samples spiked with known masses and were seen to be negligible or absent.

Figure 13 illustrates a reduction in TP of 47% and 52% for modules ZW_1 and ZW_2 , respectively.

4.4.3.3 Sulphide

No duplicates samples were prepared as all of sample volume was used undiluted to obtain sufficient sulfide to measure volumetrically by titration. Figure 13 illustrates a reduction in total sulphide of 73% and 85% for modules ZW_1 and ZW_2 respectively. Aeration of the membrane modules with normal dry air at a rate of 1.7 m³/h was present.

In a study by two chemists working in the same laboratory, the standard deviation estimated from 34 sets of duplicate sulphide measurements was 0.04 mg/L for concentrations between 0.2 and 1.5 mg/L. The average recoveries of known additions were 92% for 40 samples containing 0.5 to 1.5 mg/L and 89% for samples containing less than 0.1 mg/L (APHA 1999).

4.4.4 Microbiological Examination

4.4.4.1 Fecal Coliform

There was no evidence of fecal coliform bacteria passing the ZeeWeed[®] 500 membrane and contaminating the permeate. Initial FC concentrations in the 20% diluted SWSN

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(D₂₀) process water were 103,000 and 280,000 CFU/100 mL for ZW₁ and ZW₂ runs. To be in the required 20 to 80 range suitable for enumerating CFU's on filters used a 10^{-2} dilution would have been most suitable but was not prepared in ZW₁ run and had to be enumerated on 10^{-1} dilution. Figure 13 illustrates this 5 log removal of fecal coliforms.

4.4.5 Analysis of Metals

A general scan of metals was completed for a grab sample of process water and permeate taken half way through the system run of ZW_1 in 30% SWSN (D₃₀). Table D10, Appendix D provides a subset of a complete metals analysis. Concentrations in the dilute SWSN or permeate were not elevated to a level causing undue concern according to the livestock guidelines provided in CWQG (Task Force on Water Quality Guidelines (Canada) Council of Resource and Environment Ministers 1987). In general, a greater reduction was seen among higher valence metals and may be explained by a coagulation process or reduced ease in passing through membrane pores.

5. DISCUSSION

5.1 Evaluation of Data

5.1.1 Membrane Flux

In any baseline D_0 run a consistent linear relationship between the flow rate produced from the membrane and the suction pressure applied from the pump was observed with either membrane module both before and after filtration of dilute SWSN. Slope of the relationships varied from 0.089 to 0.16 (175% increase). The trend of this slope on these three dates is opposite for the two modules used. Figure C1, Appendix C shows the maximum and minimum slopes for the lines produced with the least squares method. All R^2 values for these runs were 0.978 (ZW₁ After1) or greater. This can be also seen in Figure 4 where the specific flux is very consistent from one flow rate to the next within each module run. Therefore the validity of comparing membrane flux performance on the basis of specific flux (L h⁻¹m⁻²kPa⁻¹) is born out.

Over the four permeate flow measurements on each of the virgin membrane modules $(ZW_1 \text{ Before and } ZW_2 \text{ Before})$ there is a significant difference observed between the modules. Although an effective area of 0.047 m² is given for the ZW-1 module any two modules do not behave identically. The J₂₀ for ZW₁ Before and ZW₂ Before were 9.7 and 11.8 L h⁻¹m⁻²kPa⁻¹, respectively and the evaluation of membrane recovery after a cleaning cycle needs to be done individually.

From Figure 4 each membrane module develops its own "identity" based on the treatment experience it has had. ZW_1 specific flux gets increasingly better in baseline D_0 checks where as ZW_2 initially drops then recovers marginally. Integrity did not appear compromised from results seen in permeate analysis, which will be described later. The manufacturer's warning that flux measurements using the compact ZW-1 module are less dependable is another explanation. The shorter and fewer rigid Zeeweed[®] straws are not open to the same cleaning action or air bubble scouring as the larger units which should produce more transferable specific flux measurements. Given the highly variable

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consistency of SWSN, wastewater quality improvement and ability to sustain a consistent flux were the main target in this project. Although an absolute barrier to pathogenic microorganisms is desired, a tradeoff between quantity of permeate produced and initial raw wastewater quality exists with membrane performance and should be assessed in system design and targeting suitable wastewater streams

 J_{20} values calculated for all D_0 runs ranged from 8.43 to 14.3 L h⁻¹m⁻²kPa⁻¹. Membrane flux is significantly reduced in diluted swine waste. The specific flux of ZW₁ and ZW₂ was reduced 14.6% and 9.3%, respectively, of the original baseline flux measured in DI.

5.1.2 Cycle Time Between Backflushes

The setting used for the backflush flow and duration were initially determined through trial and error starting from recommended values for the module. These same control inputs were used in D₉, D₂₀, and D₃₀, however they were checked operationally at the beginning of each run cycle and found to be consistent. Once set, the DAQC system allowed the computer to control the cycle without exceeding maximum value set points. As a result, cycle time between backflushes became the more definitive indicator of the level of membrane fouling. In the more dilute 9% SWSN (D9) runs, backflushes were not called for during operating period but the flux was reduced to 1.12 and 1.25 L $h^{-1} m^{-2}$ kPa^{-1} for ZW₁ and ZW₂, respectively, by the end of cycle. A forced backflush routine may have assisted performance. Figure 5 and Figure 6 for the cycle times during the D_{20} run show that the membrane were better able to sustain a stable operation during the 10 L permeate production test, however, ZW₂ in Figure 6 is approaching the minimum 3 minute threshold used. Figure C8 in Appendix D illustrates a more erratic operation in the D_{30} run and the membrane system was unable to operate consistently at or above the threshold. ZW₂ in the D₃₀ run produced more consistent results as illustrated by Figure C9 in Appendix D but cycle time was decreasing more rapidly.

5.1.3 Effect of Aeration on Membrane Flux

Aeration of the membrane provides a reduction in fouling behavior and a capacity of up to $1.7 \text{ m}^3/\text{hr}$ was recommended and practiced in the bench top design. An unplanned reduction in airflow shown in Figure C8 was indicated for the decrease in cycle time. Figure 5 shows an increase in cycle time when air tanks were changed. It is suspected but not confirmed that the airflow regulator was not reset to $1.7 \text{ m}^3/\text{hr}$ as before and an increased aeration rate explains the increased cycle time. The advantages of increased above recommended rates or variable aeration rate are worth investigation, however larger modules with more flexible membrane configurations should respond to air scouring better.

5.1.4 Understanding Membrane Fouling

Figure 7 provided a close up of a typical membrane cycling routine and illustrates the positive control that can be exerted on the membrane system utilizing a computerized DAQC system. The "quick vacuum pressure build" is more an artifact of the pump and tubing than the membrane resistance to permeate flow. The gradual and similar slope before and after the "plateau" indicated a consistent build up of foulant on the membranes. A characteristic plateau seen in D_{20} and D_{30} runs, lasting approximately 1 minute, may indicate a three stage process to fouling and is illustrated in Figure 14. In general terms, one explanation could be is as follows:

- in step 1, smaller pores are obstructed at surface of membrane with particles greater than the nominal pores size causing the gradual build in negative trans membrane pressure;
- in step 2, a small plateau occurs while smaller particles pass through largely unobstructed but build deeper in the membrane along the surface of pathways due to their forced proximity to one another; and
- in step 3, eventually the pathways are narrowed to the point medium sized particles are obstructed and pressure gradually builds to the point a backflush is required to remove these obstructive particles.

A more gradual decline in flux performance over the 10 L permeate production is more likely caused by organic fouling. Treatment and storage in 1000 and 200 mg/L NaOCl, respectively, targets this fouling as indicated by baseline measurements taken after treatment of dilute swine wastes. The level of recovery to baseline normalized flux measurements was substantial but not necessarily complete or predictable as illustrated in Figure 4.



Figure 14 Three Step Process to Membrane Fouling

5.1.5 Effect on Turbidity, TSS, TDS, COD and Conductivity

Turbidity increased in the process tank over the sample run as TSS was excluded from the permeate during the run. This trend was slowed by the replacement of diluted SWSN from the feed tank. Assuming complete mixing by aeration provided and that most turbidity would reflect the TSS concentration a 15% increase in TSS corresponded to the 33% and 27% increase in turbidity observed for ZW₁ and ZW₂, respectively.

Even dilute SWSN was highly turbid and would expect to interfere with disinfection or microbial reduction processes requiring low turbidity. Turbidity and TSS were reduced close to zero in the permeate produced. Figure 15 and Figure 16 illustrates the reductions in this membrane treatment of 20% SWSN. Table D3, Appendix D provides descriptive statistics for percentage reductions calculated for TSS, TDS, COD, turbidity and conductivity that were taken at regular intervals during the D₂₀ runs. Confidence intervals (CI_{95%}) were lowest for turbidity indicating more consistent results. CI _{95%} were highest in conductivity and statistics did not indicate a significant treatment effect on conductivity.

TDS and COD reduction was consistent in magnitude. TDS was reduced 19.1 and 26.0 % for ZW₁ and ZW₂ modules, respectively. COD was reduced 31.5 and 32.3% for ZW₁ and ZW₂ modules, respectively. As well, the TDS/COD ratios varied little within the process or permeate volumes being sampled during the run. In the D₂₀ run, this ratio was consistent as can be seen in Table D4, Appendix D. This consistent relationship was also seen in the D₉ and D₃₀ runs for matched TDS and COD samples taken. This observation indicates that the less laborious COD analysis can be effectively used for system monitoring for dilute SWSN (<30%) once the relationship is determined. Conductivity, which is often used as an estimation of TDS, did not show this relationship and was changed little by membrane treatment. This is explained in that the membrane primarily removes suspended solids, which are not a major contributor to ionic strength.



Figure 15 TSS, TDS, COD, Turbidity and Conductivity Reductions for D₂₀ Run of ZW₁



Figure 16 TSS, TDS, COD, Turbidity and Conductivity Reductions for D₂₀ Run of ZW₂

5.1.6 Biochemical Oxidation Demand

Reductions of BOD as illustrated in Figure 12 were 24 and 33% for ZW_1 and ZW_2 modules, respectively. However, the values are still very high and anoxic conditions could readily result in an enclosed vessel or pipeline without adequate aeration. There is ample nutrient for anaerobic bacteria, including sulfur reducing bacteria that may be present or introduced. Negative effects on odour, taste, and biofilm growth could be excessive or hard to control.

5.1.7 Inorganic Non-metallic Constituents (N, P, and Sulphide)

There was little reduction in ammonia as would be expected with microfiltration of N species in ionic form. The difference between TKN and NH_3 results on the same sample can be attributed to organic N in this wastewater. Its removal parallels reductions in TSS of which organic N would be commonly bound with.

The 47% and 52% reduction in TP during microfiltration by the ZeeWeed[®] 500 membrane suggests that at least half of the phosphate present is bound to suspended solids, even in a dilute SWSN with aeration. TSS were reduced to zero in this process.

Sulphide was reduced by an average of 79% in the membrane system. The pH present was in the 7.7 to 8.15 range, which also marks the transition between ionized, HS^- and S^{2-} above pH 8 and un-ionized H₂S species below pH 8 (Sawyer, McCarty and Parkin 1994). Aeration of the membrane would also expect to volatilize much of the gaseous H₂S produced. Under conditions and time frames conducive to anaerobic bacteria, the sulphates present in SWSN, would be transformed into odourous and potentially hazardous sulphides.

5.1.8 Effect on Microbial Populations

The ZeeWeed[®] 500 membrane used in the ZW-1 module was an effective barrier to one indicator of microbial contamination, fecal coliforms. Other indicator organisms including pathogenic bacteria, enteric viruses, fungi, or pathogenic protozoa were not tested. However, given the evidence of a complete barrier to suspended solids and FC,

organisms larger than FC should be excluded as long as membrane integrity and crosscontamination control was maintained.

5.2 Comparison With Results Reported in the Literature

Performance comparisons within the same membrane type and between membrane types need to take into account changing flux with operating pressure, temperature and effective membrane area. Specific operating requirements to reduce fouling and maintain membrane integrity are also system specific. The calculation of a normalized specific flux aided this comparison. Natural, non synthetic wastewaters like SWSN are highly variable and very few examples of utilizing membrane treatment on this waste stream were found in the literature. However, for illustration purposes, one example using membrane treatment of sow slurry is shown.

An inorganic silicon-carbide membrane (cutoff of 0.05 μ m, filter area of 0.05 m²) was tested for ultrafiltration of pig slurry and other agricultural wastewaters along with other membrane types (Reimann and Yeo 1997). After 6 hours of operating this membrane at 200 kPa the permeate flux had leveled out to 85 L h⁻¹m⁻². A temperature was not reported but values are assumed to be temperature corrected given capacity within the system for cooling of the feed tank. At the optimum pressure of 200 kPa produced a COD rejection of 79% was reported. Reinmann's ultrafiltration membrane produced a permeate flux of 0.42 L h⁻¹m⁻²kPa⁻¹ (after 6 hours). This compared to an average of 1.36 L h⁻¹m⁻²kPa⁻¹ at 20 °C (1.30, 1.42) for the ZW-1 modules (nominal pore diameter 0.04 μ m) where a 32% COD reduction occurred. The normalized specific flux was 3.2 times greater for the ZW-1 module on 20% SWSN and could indicate a significant difference but comparisons between treatment studies with different membrane configurations and raw waters that exhibit great variability must be interpreted carefully, if at all. Other measurements such as fouling behavior, cleaning characteristics, and pretreatments required will be equally important.

5.3 Full Scale System Design Example

Using the results of from the best run operation of the ZW-1 membrane module in 20% SWSN (D_{20}), an estimate of the membrane sizes required was made to match the waste

production from grower-finisher (feeder) pigs ranging from 500 to 10,000 sow pig production. The calculations are shown for a 1,000 sow size, which would have an estimated 6192 feeder pigs in the barn at any one time. (See Appendix E - System Design Calculations). A membrane size of 225 m² or $0.036 \text{ m}^2/\text{pig}$ was estimated and would require 80% recycle or dilution water to maintain a suitable strength process water. No redundancy was built into this estimate. In volume, an estimated 18.9% of water needs could be supplied.

There are significant assumptions made in producing this membrane sizing example and a detailed analysis of them was not part of this project which concentrated on the performance characteristics of the hollow immersed membrane in swine waste supernatant. They are identified and discussed below.

The scaled up membrane size would produce similar performance characteristics to the compact ZW-1 membrane. From manufacturer's literature, less fouling would be expected due to the increased effectiveness of air bubble scouring and the physical abrasion of the membrane straws against each other. This should increase cycle duration, increase flux, and thereby reduce the size of membrane required.

The quality of permeate produced could be recycled for the water needs of the swine facility. Although the ZeeWeed[®] 500 membrane may be an effective barrier to microbial populations as was indicated by complete removal of fecal coliforms, virus removal was not assumed. Turbidity was also greatly reduced and would assist in the application of the effective application of other unit treatment processes such as disinfection by UV or microbial reduction with chlorine. However the permeate was still nutrient rich and without reduction or system protection would be open to microbial contamination and regrowth.

Membrane integrity and maintenance could be sustained in the harsh environment of a swine facility and wastes generated from it. There was not evidence of membrane integrity failure during the bench top test runs. The hydraulic and microfiltration performance characteristics of a hollow, immersed membrane, as indicated by the Zenon ZW-1 module, produced results consistent with manufacturer claims.

The cost of applying an advanced treatment process, either individually or in combination with other unit processes, would have to be sustained within the economic structure of a modern swine operation. Only a small amount of financial cost or operator time can typically be applied to the operation of a waste management system. In areas where hog farms are not already constrained by land or water resources, the advantage of swine waste treatment over land applied waste as a organic crop fertilizer could be difficult to realize. Other parallel benefits such as biogas production or volume reduction to reduce spreading costs may be possible. In a recent study focusing mainly on South Carolina conditions, only \$1.30 US per hog sold was used as the cost criterion whether a particular manure storage and treatment system was viable for swine finishing farms (Chastain, et al 2002).

An estimate of the membrane sizing required for various sizes of swine feeder operations and resulting waste production in Table 4.

Breeding	Feeder	Waste	Water Needs	Membrane
Sows	Pigs	Production	(L/day)	Size (m ²)
		(L/day)		
500	3,096	24,956	21,084	113
1,000	6,192	49,911	42,168	225
3,000	18,577	149,733	126,504	675
5,000	30,962	249,955	210,840	1,125
10,000	61,923	499,110	421,680	2,250

 Table 4
 Membrane Sizing Estimate for Swine Feeder Facilities

6. CONCLUSIONS

The ZW-1 compact membrane module can be used for preliminary investigation into the performance of commercial ZeeWeed[®] 500 modules in highly polluted livestock wastewaters as evidenced by a direct filtration of diluted swine waste supernatant. A data acquisition and control system developed was very useful in controlling the direct filtration process when operating close to fouling limits in highly contaminated wastewater. Control based on trans-membrane pressure provided excellent results. Cycle time was a good indicator to monitor membrane fouling. In terms of fouling behavior, the performance results obtained from one compact ZW-1 module should not be assumed for another and must be treated and tested individually.

Membrane performance based on normalized specific flux provided an excellent method of performance comparison. Compared to baseline measurements in distilled water, the specific flux of a Zeeweed[®] 500 compact membrane module was significantly reduced in diluted swine waste supernatant (SWSN). In the best run in 20% SWSN (average COD 2.7 g O₂/L), a flux 1.36 L h⁻¹m⁻²kPa⁻¹ was maintained over a 10 L permeate production period. The specific flux of ZW₁ and ZW₂ was reduced 14.6% and 9.3% of the original baseline flux measured in DI.

Direct membrane treatment was an effective and a compete barrier to fecal coliforms and suspended solids and as expected, allows dissolved components to pass easily. Turbidity reduction was excellent and would allow microorganism reduction processes to work more effectively. The greatest turbidity reduction measured was for 30% SWSN at 646 NTU to ≤ 0.5 NTU.

Reduction in percentage terms of other key contaminants, from highest to lowest was sulphide (79%), TP (49.5%), TOC (34%), COD (32%), BOD₅ (29%), TDS (23%), TKN (21%), and NH₃ (5.9%). These were are calculated from simple average of two modules but were fairly consistent and did not relate strongly to hydraulic performance. Soluble organic and inorganic materials passed through the membrane and would need further

reduction for potable reuse options. Swine wastewater treated by direct microfiltration will still be rich in nutrients by municipal standards and quality can be impaired quickly. Pretreatment by sedimentation and dilution made direct membrane treatment possible.

Small size membrane testing provided good qualitative results, however great caution should be used when scaling up the results. Based on specific flux generated from this project, a preliminary membrane size was calculated for the waste production generated from 6192 feeder pigs that could be produced from a 1000 sow breeding herd. A membrane size of 225 m² or 0.036 m²/pig was estimated and would require 80% recycle or dilution water to maintain suitable strength process water. An estimated 18.9% of water needs could be supplied to the feeder pig operation.

From review of the literature, direct potable reuse has little policy support, and is undeveloped for potable reuse for animals watering. At this stage only indirect potable reuse is viewed as a viable application of reclaimed water, but only where there is a careful, thorough, project-specific assessment that includes contaminant monitoring, health and safety testing, and system reliability evaluation. On a project specific basis, water conservation techniques and matching agricultural production to water resources available should be the first step in evaluating appropriate technologies for water needs of an intensive livestock operation. The financial and labor realities existing in intensive livestock will be stressed to utilize the technical and management practices required for advanced wastewater treatment and reuse.

7. RECOMMENDATIONS

Hollow fiber immersed membranes can be a positive barrier to microbiological contamination and could enable microorganism reduction processes to work better. Their combination with other unit processes should be investigated. Future studies targeting membrane performance should assess this tradeoff between quantity and quality. Other microbiological indicators such as presence/absence test for E. *coli* could be easily added to the evaluation.

A small amount of air was manually bled from the system on the permeate side of the membrane. An automated air bleed should be incorporated into larger systems. Measurement of sustainable flux should also address temperature effects over a wider range and the effect and economics of increase aeration in polluted wastewaters.

Surrogate contaminants to utilize in place of raw swine waste should be investigated for future bench top experiments. Larger membrane modules should provide better and more transferable flux measurements for designing field scale operations, if appropriate. A pilot scale study over a longer time period would be required.

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APPENDIX A – SENSOR CALIBRATION

- Figure A1 Calibration of Pressure Transmitter (EW 68848-26) at 9 volts
- Figure A2 Calibration of Pressure Gauge
- Figure A3 Calibration of T1 Process Tank Thermistor
- Figure A4 Calibration of T2 Permeate Tank Thermistor



Figure A1 Calibration of Pressure Transmitter (EW 68848-26) at 9 volts



Figure A2 Calibration of Pressure Gauge



Figure A3 Calibration of T1 Process Tank Thermistor



Figure A4 Calibration of T2 Permeate Tank Thermistor

APPENDIX B – SYSTEM COMPONENTS

- Table B1System Equipment List
- Figure B1 System Control Screen
- Figure B2 System Design Wire Diagram
- Figure B3 Visual Basic 6.0 Design Code

	Manufacturer or supplier	Part # or Descriptor	Range	Resolution	Calibration
Data Acquisition and Contro	ol System	L	I	1	
Compound Pressure Transmitter	Cole Parmer	EW-68848-26	-14.7 to 30 psig, 1 to 5 V Output	<u>+</u> 1% full scale	Druck DPI 1603
Pressure snubber	Badger Eng.	1/4" NPT	· · · · · · · · · · · · · · · · · · ·		1
Compound pressure gauge (visual)	Cole Parmer	EW-68950-00	-30"Hg, to +99.9 psi	0.1 psi	Druck DPI 1603
Thermistors, NTC 5000 ohms @ 25°C	Thermometrics	RL1003-2871- 103-SA	-50 to 150 °C	±1℃	manual
Computer	IBM	Pentium 2	n/a	n/a	n/a
Analog & Digital I/O Board	Measurement Computing Corp. (Dycor)	PCI-DAS1000	+ 10 V, 250 kHz, 8 diff. channels	12 bits	n/a
Relay	Dycor		50 pin	n/a	n/a
DC Voltage source	BK Precision	1760A	0 to 30 V	millivolts	Fluke Multimeter
DAQC software	Measurement Computing Corp. (Dycor)	Softwire 3.1	n/a	n/a	n/a
Programming software	Microsoft	Visual Basic 6.0	n/a	n/a	n/a
Membrane and Pumping Sy	stem	1			<u> </u>
Masterflex L/S Drive c/w RS- 232	Cole Parmer	EW-07550-20 EW-07550-64	1.6 to 100 rpm	<u>+</u> 0.25% F.S.	Built in
2- ZW-1 Bench Test Units	Zenon Environmental	ZW500d©		n/a	n/a
2 – Easy Load Pump Heads	Cole Parmer	EW-07518-12	L/S 15 or L/S 24 tubing	n/a	manual
High- Performance Precision tubing (Tygon)	Masterflex	BH-06429-15 EW-06429-24	2.6 to 238 mL/min (c/w 1.6 to 100 rpm drive)	n/a	manual
Polyethylene (LDPE) tubing	Cole Parmer	A-95626-03	11/64" ID x ¼" OD	n/a	n/a
Fittings and adapters (assorted)	John Guest, plus Swagelok (for ¼" OD LDPE tubing)	EW-06360-05 EW-06360-44 EW-06384-20 A 06372-01 A- 06372-10	1/8" to 1/4"	n/a	n/a
Aeration System					
2 Air Extra Dry	Prax Air	T size cylinder	Class 2.2 UN1002	n/a	n/a
Tank T and pigtail	Prax Air	WES T93 WES PF2-4-24	CGA 590 T 24" pigtail	n/a	n/a
Dual stage cylinder regulator	Prax Air	CDN 412-3331- 350	0-2500 psi 0 to 100 psi	n/a	n/a
Air flowmeter	Dwyer		0-100 scfh	n/a	Wet Test Meter GCA/Precision Scientific (63115)

Table B1 System Equipment List



Figure B1 System Control Screen



Figure B2.1 System Design Wire Diagram - Pump Control



Figure B2.2 System Design Wire Diagram – A/D Control (Sensor Reading)



Figure B2.3 System Design Wire Diagram – Sensor Conversion and Display



Figure B2.4 System Design Wire Diagram - Excel Control

```
Form1 - 1
Public StringToPump As String
Public PumpRPM As Single
Public OrgPumpRPM As Single
Public TankVolume As Double
Public Pressure As Double
Public Backflush As Boolean
Public CycleCount As Integer
Public CycleTime As Integer
Public CurrentCycleTime As Integer
Public NCyclesLT5Min As Integer
Public PumpOn As Boolean
Public Pause As Boolean
Private Sub Form_Load()
' SoftWIRE
' IMPORTANT - The following lines of Load code were added by SoftWIRE.
            Do not remove them or the program will not run correctly.
Load Form1
Wirel.Initialize
'Set Variables when program is loaded
   Backflush = False
   Pause = False
End Sub
Private Sub Form_QueryUnload(Cancel As Integer, UnloadMode As Integer)
 * ***********
' SoftWIRE
' IMPORTANT - The following lines of QueryUnload code were added by SoftWIRE.
            Do not remove them or the program will not run correctly.
Dim Form As Form
Dim Count As Integer
On Error Resume Next
Count = 0
For Each Form In Forms
   If Form.Visible Then
       Count = Count + 1
   End If
Next Form
If Count > 1 Then
   Cancel = True
   Me.Hide
Else
   For Each Form In Forms
      Unload Form
   Next Form
End If
End Sub
Private Sub SerialOut1_RunBlock()
'Routine to display the command string being sent to the pump.
   lblStringToPump.Caption = StringToPump
End Sub
Private Sub CBDivide1_RunBlock()
'Displays temperature T1
   txtTemperature1.InputText = Format(CBDivide1.Value, "##0.0")
End Sub
```

Figure B3 Visual Basic 6.0 Design Code

```
Form1 - 2
 Private Sub CBDivide2_RunBlock()
 'Displays pressure P1
     Pressure = CBDivide2.Value
     txtPressure.InputText = Format(CBDivide2.Value, "##0.000")
End Sub
Private Sub CBDivide3_RunBlock()
 'Displays temperature T2
     txtTemperature2.InputText = Format(CBDivide3.Value, "##0.0")
End Sub
Private Sub Timer1_StatusReady()
 'Ref. 1. Routine to determine pump rates and limits for 'each time interval of timer1 (set to 1 second)
     Dim PumpFlowRate As Single
     If PumpOn = True Then
           Converts pump RPM to ml/min flow rate based on input conversion
          'from calibration.
          PumpFlowRate = PumpRPM * 2.455
           'Calculate tank volume
          TankVolume = TankVolume + PumpFlowRate / 60000#
           'Calculate cycle time.
          CycleTime = CycleTime + Timer1.Interval / 1000
          'Display Pump RPM, Flow rate and tank volume at each time interval.
txtPumpRPM.InputText = PumpRPM
txtFlowRate.InputText = PumpFlowRate
          txtTotalVolume.InputText = Format(TankVolume, "#0.0000")
      ' Test if tank volume > 10L or < 0, if so, quit in routine Halt.
          If TankVolume >= 10# Then
Halt "Tank Volume > 10 Litres."
          End If
          If TankVolume <= 0# Then
Halt "Tank Volume < 0 Litres."
          End If
      'Test if Pressure is out of range, if so, Pause in routine Paused
If Pressure < -9# Or Pressure > 9# Then
Paused "Pressure out of range."
          End If
     End If
End Sub
Private Sub Halt (Msgs As String)
'Routine to stop pump, cancel all timers, and print message.
StringToPump = Chr(2) & *P01H* & Chr(13)
          PumpRPM = 0
          SerialOut1.OutputString = StringToPump
          Timer1.CancelBlock = True
          Timer2.CancelBlock = True
          Timer3.CancelBlock = True
          PumpOn = False
          Message Msgs
End Sub
Private Sub Paused (Msgs As String)
 'Routine to print a pause message and calls the PauseExperiment routine.
     PauseExperiment
 Msg = "Program Paused because " & Msgs & "Press OK, fix problem and then Continue to continue
running experiment"
Style = vbOKOnly
Title = "Experiment Paused"
     Response = MsgBox(Msg, Style, Title)
End Sub
```

Figure B3 Visual Basic 6.0 Design Code (cont'd)

```
Form1 - 3
Private Sub Message(Msgl As String)
'Routine to print a halt message
     Dim Style, Title, Response
     Msg = "Program Halted because " & Msg1
     Style = vbOKOnly
Title = "Experiment Halted"
     Response = MsgBox(Msg, Style, Title)
End Sub
Private Sub cmdInitPump_RunBlock()
 'Ref. 2 Command Button which sends initaliazion command to pump
StringToPump = Chr(5) & "P?2" & Chr(13)
SerialOutl.OutputString = StringToPump
End Sub
Private Sub cmdPause_RunBlock()
'Ref. 3. Command Button which pauses the timer.
     If Pause = False Then
          PauseExperiment
     Else
          Allows flowrate reset on Pause
          OrgPumpRPM = txtInitRPM.InputText
          PumpRPM = OrgPumpRPM
          ContinueExperiment
     End If
End Sub
Private Sub PauseExperiment()
  Ref. 3. Routine that pauses the experiment and stops the pump.
     PumpOn = False
Pause = True
     Timer1.TimerEnabled = False
     Timer2.TimerEnabled = False
Backflush = False
     Timer3.TimerEnabled = False
     cmdPause.Caption = "Continue Exp."
StringToPump = Chr(2) & "P01H" & Chr(13)
SerialOut1.OutputString = StringToPump
End Sub
Private Sub ContinueExperiment()
 Ref. 3. Routine to continue the experiment after a pause.
cmdPause.Caption = "Pause Exp."
StringToPump = Chr(2) & "P01S+" & Str(PumpRPM) & "V999999G" & Chr(13)
     SerialOut1.OutputString = StringToPump
     PumpOn = True
Pause = False
Timer1.TimerEnabled = True
     Timer3.TimerEnabled = True
     Pressure = 0
     txtPressure.InputText = Format(Pressure, "##0.000")
End Sub
Private Sub cmdStop_RunBlock()
'Ref. 4. Command button to stop the experiment.
StringToPump = Chr(2) & "P01H" & Chr(13)
    SerialOut1.OutputString = StringToPump
    PumpRPM = 0
   PumpOn = False
Timer1.Break = True
    Timer2.Break = True
End Sub
Private Sub cmdStartPump_RunBlock()
'Ref. 5. Command Button to start the pump in the direction and at the RPM
'as indicated in the text box txtInitRPM.
     If txtInitRPM.Text > 0 Then
```

Figure B3 Visual Basic 6.0 Design Code (cont'd)

```
Form1 - 4
         PumpOn = True
         StringToPump = Chr(2) & "P01S+" & Str(Abs(txtInitRPM.Text)) & "V99999G" & Chr(13)
    Else
         StringToPump = Chr(2) & "P01S-" & Str(Abs(txtInitRPM.Text)) & "V99999G" & Chr(13)
         PumpOn = True
    End If
    SerialOut1.OutputString = StringToPump
    OrgPumpRPM = txtInitRPM.InputText
    PumpRPM = txtInitRPM.InputText
     Allows addition of data to same sheet after Pause without overwriting
    StExcelWrite1.StartRow = ExcelStartRow.InputText
    TankVolume = 0#
    CycleCount = 0
    CycleTime = 0
    txtCycleCounter.Text = CycleCount
    NCyclesLT5Min = 0
    Backflush = False
End Sub
Private Sub cmdPumpStatus RunBlock()
Ref. 6. Command Button to ask pump for status.
StringToPump = Chr(2) & "POIC" & Chr(13)
   SerialOut1.OutputString = StringToPump
End Sub
Private Sub cmdRPMStatus RunBlock()
'Ref. 7. Command Button to ask pump for RPM.
StringToPump = Chr(2) & "P015" & Chr(13)
    SerialOut1.OutputString = StringToPump
End Sub
Private Sub cmdZeroStatus_RunBlock()
'Ref. 8. Command button to set the zero status of the pump.
StringToPump = Chr(2) & "P01ZZ0" & Chr(13)
SerialOut1.OutputString = StringToPump
End Sub
Private Sub Timer2_RunBlock()
'Ref. 9. Routine that displays timer2 interval
    txtInterval = Timer2.Interval
End Sub
Private Sub Timer2_StatusReady()
'Ref. 9. Timerz controls the backflushing cycles. This routine keeps
'track of the number of backflush cycles that have been performed and
'halts if the cycle count limit is exceeded.
    Dim CycleTimeLimit As Integer
      CycleTimeLimit = CInt(txtCycleTime.Text) * 60
     CycleCount = CycleCount + 1
txtCycleCounter.Text = CycleCount
' if cycle time < cycle time limit input add one to
' number of cyles less than time limit.
' else set to zero.
      If CurrentCycleTime < CycleTimeLimit Then
NCyclesLT5Min = NCyclesLT5Min + 1
       Else
           NCyclesLT5Min = 0
       End If
       CycleTime = 0
    If NCyclesLT5Min >= Val(txtCycleCount.Text) Then
        Halt "Cycle Count Limit exceeded"
    Else
' Allows reset of flowrate after counter limit exceeded
         OrgPumpRPM = txtInitRPM.InputText
         PumpRPM = OrgPumpRPM
```

Figure B3 Visual Basic 6.0 Design Code (cont'd)

```
Form1 - 5
        StringToPump = Chr(2) & "P01S+" & Str(PumpRPM) & "G" & Chr(13)
         SerialOut1.OutputString = StringToPump
        Backflush = False
        Timer2.CancelBlock = True
    End If
End Sub
Private Sub cmdBackFlush_RunBlock()
'Ref. 9. Command button to manually perform backflushing.
    Backflush = True
    PumpRPM = -Abs(txtBackFlushRPM.InputText)
    StringToPump = Chr(2) & "POIS" & Str(PumpRPM) & "G" & Chr(13)
SerialOutl.OutputString = StringToPump
    Timer2.TimerEnabled = True
End Sub
Private Sub CBUnpack2_RunBlock()
'Ref. 10. Defines and applies pressure data filter
    Dim myarray2() As Single, myarr() As Single
    Dim i As Integer, ii As Integer, U As Integer, L As Integer
    ReDim myarr(200)
    myarray2 = CBUnpack2.OutputArray
U = UBound(myarray2)
    L = LBound(myarray2)
    ii = -1
    i = -1
    Do
    i = i + 1
    If myarray2(i) > 1# And myarray2(i) < 3.7 Then
        ii = ii + 1
    myarr(ii) = myarray2(i)
End If
    Loop Until i = U
    lblNumber.Caption = ii + 1
    If ii < 199 Then ReDim Preserve myarr(0 To ii)
    Statistics1.Value = myarr
End Sub
Private Sub User1_RunBlock()
 Ref. 10. Puts Pressure, flowrate and tank volume into MyArray at each
' time interval as required by CBStripchart block.
    Dim MyArray(2, 0) As Double
    MyArray(0, 0) = Pressure
    MyArray(1, 0) = txtFlowRate.Text
MyArray(2, 0) = TankVolume
    User1.Value = MyArray
End Sub
Private Sub cboTimerInterval_RunBlock()
'Ref. 11. Settings for real-time charting for P, F, and TV
    Dim T As Integer
    T = Val(cboTimerInterval.Text)
    CStripChart1.XSpacing = T / 60
    If T = 5 Then
        CStripChart1.XAxis.LinearScale.MaxValue = 5
        CStripChart1.XAxis.MajorTick = 4
        CStripChart1.XAxis.MinorTick = 11
    End If
If T = 10 Then
        CStripChart1.XAxis.LinearScale.MaxValue = 10
        CStripChart1.XAxis.MajorTick =
        CStripChart1.XAxis.MinorTick = 5
    End If
    If T = 30 Then
```

Figure B3 Visual Basic 6.0 Design Code (cont'd)

```
Form1 - 6
           CStripChart1.XAxis.LinearScale.MaxValue = 30
           CStripChart1.XAxis.MajorTick = 29
           CStripChart1.XAxis.MinorTick = 1
     End If
If T = 60 Then
           CStripChart1.XAxis.LinearScale.MaxValue = 60
           CStripChart1.XAxis.MajorTick = 11
CStripChart1.XAxis.MinorTick = 4
      End If
End Sub
Private Sub Timer3_StatusReady()
'Ref. 12. Timer3 controls the rate at which the AnaloginScan block obtains
'readings from the pressure and temperature sensors.
'Decreases the pump RPM by 4 rpm when the pressure is less than -7
     If PumpOn = True Then
If Pressure < -7# And Backflush = False Then
PumpRPM = PumpRPM - 4
'If the pump RPM goes below 1/2 of original RPM, a backflush flag is set 'to true which activates timer2 and the backflush cycle.
           If PumpRPM <= 0.5 * OrgPumpRPM Then
                      CurrentCycleTime = CycleTime
                      txtTest.Text = CurrentCycleTime
                      Backflush = True
PumpRPM = -Abs(txtBackFlushRPM.InputText)
                      StringToPump = Chr(2) & "P01S" & Str(PumpRPM) & "G" & Chr(13)
SerialOut1.OutputString = StringToPump
                      Timer2.TimerEnabled = True
                Else
                   reduce speed of pump when not < 1/2 original rpm
                      txtPumpRPM.InputText = PumpRPM
StringToPump = Chr(2) & "P01S+" & Str(PumpRPM) & "G" & Chr(13)
SerialOut1.OutputString = StringToPump
                 End If
           End If
     End If
End Sub
```

Figure B3 Visual Basic 6.0 Design Code (cont'd)
APPENDIX C – PERFORMANCE CHARTS FOR SYSTEM RUNS

- Figure C1 Linear Relationship of Suction Pressure Applied to Flow Rate
- Figure C2 Performance Chart for D9 Run of ZW1
- Figure C3 Performance Chart for D9 Run of ZW2
- Figure C4 Performance Chart for D20 Run of ZW1
- Figure C5 Performance Chart for D20 Run of ZW2
- Figure C6 Performance Chart for D30 Run of ZW1
- Figure C7 Performance Chart for D30 Run of ZW2
- Figure C8 Cycle Time for D30 Run of ZW1
- Figure C9 Cycle Time for D30 Run of ZW2



Figure C1 Linear Relationship of Suction Pressure Applied to Flow Rate



Figure C2 Performance Chart for D₉ Run of ZW₁



Figure C3 Performance Chart for D₉ Run of ZW₂



Figure C4 Performance Chart for D₂₀ Run of ZW₁



Figure C5 Performance Chart for D₂₀ Run of ZW₂



Figure C6 Performance Chart for D₃₀ Run of ZW₁



Figure C7 Performance Chart for D₃₀ Run of ZW₂



Figure C8 Cycle Time for D₃₀ Run of ZW₁



Figure C9 Cycle Time for D_{30} Run of ZW_2

ZW DO Data Analysis	All Data	ZW1	ZW2	All Before	All After1	All After2
Mean	10.9369	11.8158	10.0580	10.7537	10.0875	11.9696
Standard Error	0.3847	0.5682	0.3943	0.3992	0.5610	0.8510
Median	10.4180	11.4837	9.6807	10.7286	9.7989	11.9949
Mode	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A
Standard Deviation	1.8845	1.9683	1.3660	1.1290	1.5868	2.4071
Sample Variance	3.5513	3.8740	1.8660	1.2747	2.5180	5.7941
Kurtosis	-0.7288	-1.7193	-1.5364	-2.3725	-2.1089	-2.7488
Skewness	0.6001	0.2337	0.4321	-0.0265	0.2674	-0.0058
Range	5.9087	4.9657	3.5731	2.6301	3.8414	4.8491
Minimum	8.4331	9.3761	8.4331	9.3761	8.4331	9.4927
Maximum	14.3419	14.3419	12.0062	12.0062	12.2745	14.3419
Sum	262.4863	141.7901	120.6962	86.0299	80.6997	95.7566
Count	24.0000	12.0000	12.0000	8.0000	8.0000	8.0000
Confidence Level(95.0%)	0.7958	1.2506	0.8679	0.9439	1.3266	2.0124
					· · · · · · · · · · · · · · · · · · ·	
	ZW1 Before	ZW1 After1	ZW1 After2	ZW2 Before	ZW2 After1	ZW2 After2
	ZW1 Before	ZW1 After1	ZW1 After2	ZW2 Before	ZW2 After1	ZW2 After2
Mean	ZW1 Before 9.7260	ZW1 After1 11.5077	ZW1 After2 14.2139	ZW2 Before 11.7815	ZW2 After1 8.6672	ZW2 After2 9.7253
Mean Standard Error	ZW1 Before 9.7260 0.1485	ZW1 After1 11.5077 0.3412	ZW1 After2 14.2139 0.0917	ZW2 Before 11.7815 0.1317	ZW2 After1 8.6672 0.0879	ZW2 After2 9.7253 0.1169
Mean Standard Error Median	ZW1 Before 9.7260 0.1485 9.7712	ZW1 After1 11.5077 0.3412 11.4837	ZW1 After2 14.2139 0.0917 14.2854	ZW2 Before 11.7815 0.1317 11.8239	ZW2 After1 8.6672 0.0879 8.7134	ZW2 After2 9.7253 0.1169 9.6807
Mean Standard Error Median Mode	ZW1 Before 9.7260 0.1485 9.7712 #N/A	ZW1 After1 11.5077 0.3412 11.4837 #N/A	ZW1 After2 14.2139 0.0917 14.2854 #N/A	ZW2 Before 11.7815 0.1317 11.8239 #N/A	ZW2 After1 8.6672 0.0879 8.7134 #N/A	ZW2 After2 9.7253 0.1169 9.6807 #N/A
Mean Standard Error Median Mode Standard Deviation	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339
Mean Standard Error Median Mode Standard Deviation Sample Variance	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965 0.6092	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225 1.4857	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346 0.3992	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929 0.5343	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717 0.3758	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556 0.5544
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965 0.6092 9.3761	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225 1.4857 10.7888	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346 0.3992 13.9427	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929 0.5343 11.4719	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717 0.3758 8.4331	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556 0.5544 9.4927
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965 0.6092 9.3761 9.9854	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225 1.4857 10.7888 12.2745	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346 0.3992 13.9427 14.3419	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929 0.5343 11.4719 12.0062	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717 0.3758 8.4331 8.8089	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556 0.5544 9.4927 10.0471
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum Sum	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965 0.6092 9.3761 9.9854 38.9039	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225 1.4857 10.7888 12.2745 46.0308	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346 0.3992 13.9427 14.3419 56.8554	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929 0.5343 11.4719 12.0062 47.1260	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717 0.3758 8.4331 8.8089 34.6690	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556 0.5544 9.4927 10.0471 38.9012
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum Sum Count	ZW1 Before 9.7260 0.1485 9.7712 #N/A 0.2970 0.0882 -3.6889 -0.3965 0.6092 9.3761 9.9854 38.9039 4.0000	ZW1 After1 11.5077 0.3412 11.4837 #N/A 0.6825 0.4658 -3.4155 0.1225 1.4857 10.7888 12.2745 46.0308 4.0000	ZW1 After2 14.2139 0.0917 14.2854 #N/A 0.1833 0.0336 3.4585 -1.8346 0.3992 13.9427 14.3419 56.8554 4.0000	ZW2 Before 11.7815 0.1317 11.8239 #N/A 0.2634 0.0694 -3.7585 -0.3929 0.5343 11.4719 12.0062 47.1260 4.0000	ZW2 After1 8.6672 0.0879 8.7134 #N/A 0.1758 0.0309 -0.6437 -0.9717 0.3758 8.4331 8.8089 34.6690 4.0000	ZW2 After2 9.7253 0.1169 9.6807 #N/A 0.2339 0.0547 1.7600 1.0556 0.5544 9.4927 10.0471 38.9012 4.0000

Table C1	Descriptive	Statistics	for	Normalized	Specific	Flux	(J_{20})	Data	for D	0 Runs
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APPENDIX D - DATA FOR WATER QUALITY ANALYSIS

- Figure D1 Standard Curve for COD Determination @600nm
- Figure D2 Standard Curve for TP Determination @470nm
- Table D1 Water Quality of Di-ionized Water (DI) for D₀ Baseline Runs
- Table D2 Water quality Over D₉ and D₃₀ Runs
- Table D3 Descriptive Statistics for Percentage Reductions in D₂₀ Runs
- Table D5 Ratio of TDS to COD for Samples Over D_{20} Run.
- Table D6Total Organic Carbon Analysis
- Table D7 5-Day Biochemical Oxygen Demand Analysis (D₂₀)
- Table D8 Total Kjedahl Nitrogen Analysis (D₂₀)
- Table D9 Ammonia Analysis (D₂₀)
- Table D10 Total Phosphorus Analysis (D₂₀)
- Table D11 Metal Scan of Effluents From D₃₀ Run of ZW₁

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Figure D2 Standard Curve for TP Determination @470nm

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ZW-1 Module	ZW ₁	ZW ₂	ZW ₁	ZW ₂	ZW ₁	ZW ₂
Date (mm/dd/yy)	05/28	3/03	11/14/03	11/18/03	1/2	6/04
рН @25°С	6.5	6.5	6.3	5.9	6.5	6.5
Conductivity (µS/cm)	n/a	n/a	40.2	32.5	45.5	45.5
Temperature (°C)	20.8	20.8	18.8	17.9	21	21

Table D1 Water Quality of Di-ionized Water (DI) for D0 Baseline Runs

Table D2 Water Quality Over D₉ and D₃₀ Runs *

	Turbidit	Conductivity	TSS	TDS	COD	Temp	pН
	y NTU	mS/cm	mg/L	mg/L	mg O ₂ /L	°C	
ZW ₁	100 /	1.73 / 1.64	38.6 /	537 /	1236 / 986	19.6	8.1
D9	0.25		0**	410			
ZW ₂	130 /	1.72 / 1.67	49.0 /	450 /	1368 / 1045	19.3	8.0
D9	0.30		0**	335			
ZW ₁	646/	8.78 / 8.42	380 /	2320 /	4854 / 3686	19.5	7.95
D ₃₀	0.50		8.0	1860			

* average over run reported (process / permeate), single values for process tank sample

** Drying of permeate samples for TSS consistently showed negative weight gains during drying (<0.5 mg). Checks with DI using the same procedure did not show these discrepancy. Results showing < 0.5 mg reported as zero.

ZW1 D20 Reduction (%)	TSS	TDS	COD	Turbidity	Conductivity
Mean	102.4	19.1	31.5	99.9	-7.7
Standard Error	0.8	2.3	1.0	0.0	5.0
Median	102.3	20.7	31.3	99.9	-4.7
Mode	100.0	#N/A	#N/A	#N/A	#N/A
Standard Deviation	2.1	5.1	2.1	0.01	11.2
Sample Variance	4.5	25.6	4.3	0.0	124.5
Kurtosis	-0.5	4.6	1.4	-0.9	4.5
Skewness	0.4	-2.1	0.5	-0.4	-2.1
Range	5.8	12.4	5.0	0.0	26.5
Minimum	100.0	10.1	29.2	99.9	-27.4
Maximum	105.8	22.5	34.2	99.9	-0.9
Sum	716.8	95.3	126.0	499.6	-38.7
Count	7	5	4	5	5
Confidence Level (95.0%)	2.0	6.3	3.3	0.01	<u>1</u> 3.9
ZW2 D20 Reduction (%)	TSS	TDS	COD	Turbidity	Conductivity
ZW2 D20 Reduction (%)	TSS	TDS	COD	Turbidity	Conductivity
ZW2 D20 Reduction (%) Mean	7SS 101.4	<i>TDS</i> 26.0	COD 32.3	<i>Turbidity</i> 99.9	Conductivity 9.0
ZW2 D20 Reduction (%) Mean Standard Error	TSS 101.4 0.5	TDS 26.0 1.4	COD 32.3 1.3	<i>Turbidity</i> 99.9 0.0	Conductivity 9.0 7.4
ZW2 D20 Reduction (%) Mean Standard Error Median	TSS 101.4 0.5 101.1	TDS 26.0 1.4 27.7	COD 32.3 1.3 31.9	Turbidity 99.9 0.0 99.9	Conductivity 9.0 7.4 6.9
ZW2 D20 Reduction (%) Mean Standard Error Median Mode	TSS 101.4 0.5 101.1 100.0	TDS 26.0 1.4 27.7 20.9	COD 32.3 1.3 31.9 35.2	Turbidity 99.9 0.0 99.9 #N/A	Conductivity 9.0 7.4 6.9 #N/A
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation	TSS 101.4 0.5 101.1 100.0 1.4	TDS 26.0 1.4 27.7 20.9 4.4	COD 32.3 1.3 31.9 35.2 2.9	Turbidity 99.9 0.0 99.9 #N/A 0.01	Conductivity 9.0 7.4 6.9 #N/A 16.6
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance	755 101.4 0.5 101.1 100.0 1.4 2.0	TDS 26.0 1.4 27.7 20.9 4.4 19.2	COD 32.3 1.3 31.9 35.2 2.9 8.4	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis	7SS 101.4 0.5 101.1 100.0 1.4 2.0 0.6	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness	7SS 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range	TSS 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1 4.3	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3 11.5	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1 6.3	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2 0.0	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4 43.4
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum	755 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1 4.3 100.0	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3 11.5 20.1	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1 6.3 29.0	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2 0.0 99.9	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4 43.4 -6.9
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum	7SS 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1 4.3 100.0 104.3	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3 11.5 20.1 31.6	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1 6.3 29.0 35.2	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2 0.0 99.9 99.9	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4 43.4 -6.9 36.5
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum Sum	7SS 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1 4.3 100.0 104.3 1014.1	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3 11.5 20.1 31.6 260.3	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1 6.3 29.0 35.2 161.4	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2 0.0 99.9 99.9 99.9	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4 43.4 -6.9 36.5 44.9
ZW2 D20 Reduction (%) Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum Sum Count	7SS 101.4 0.5 101.1 100.0 1.4 2.0 0.6 1.1 4.3 100.0 104.3 1014.1 10	TDS 26.0 1.4 27.7 20.9 4.4 19.2 -1.8 -0.3 11.5 20.1 31.6 260.3 10	COD 32.3 1.3 31.9 35.2 2.9 8.4 -2.7 0.1 6.3 29.0 35.2 161.4 5	Turbidity 99.9 0.0 99.9 #N/A 0.01 0.0 1.6 -1.2 0.0 99.9 99.9 99.9 499.6 5	Conductivity 9.0 7.4 6.9 #N/A 16.6 276.6 2.5 1.4 43.4 -6.9 36.5 44.9 5

Table D3 Descriptive Statistics for Percentage Reductions in D_{20} Runs

Permeate		TDS/COD							
Volume (L)	ZW1 PT	ZW1 IL	ZW2 PT	ZW2 IL					
1	n/a	n/a	0.41	0.43					
1	n/a	n/a	0.43	0.44					
3	0.35	0.39	0.38	0.43					
3	0.36	0.40	0.38	0.43					
5	0.32	n/a	0.44	0.45					
5	0.34	n/a	0.41	0.42					
7	0.34	0.39	0.39	0.48					
7	0.32	0.37	0.39	0.47					
9	0.30	0.41	0.43	0.46					
9	0.32	n/a	0.40	0.44					
Mean	0.33	0.39	0.41	0.44					
SDEV	0.02	0.02	0.02	0.02					

Table D4 Ratio of TDS to COD for Samples Over D_{20} Run

Table D5 Total Organic Carbon Analysis

Total Organ	ic Carbon	Analysis		Method:	5310 B						
Date	Oct 8th, 20	003		Equipment	Dohrmann	Carbon Ana	lyzer (DC-80))			
				Assisting:	Maria Dem	eter					
Sample	TC Raw	Dilution	TC	TIC Raw	Dilution	TIC	тос	Dilution	Original TOC	Reduction	Comments
Units	mg/L		mg/L	mg/L		mg/L	mg/L		mg/L	%	
TC 400 Std	388.9										KHP Std
TIC 400 Std				399.9							Na ₂ CO ₃ Std
PT8/12	587.1	0.5	1178.7	3.141	1	2.278	1176.4	0.3	3921.3		
	590.2	0.5		1.414	1						
	590.7	0.5			1						
IL8/12	473.0	0.5	947.3	0.444	1	0.503	946.8	0.3	3156.1	19.5	
	484.0	0.5		0.561	1						
	464.0	0.5			1						
PT9/9	346.4	0.5	663.0	53.56	1	51.5	611.4	0.2	3057.1		
	336.7	0.5		50.84	1						
	330.9	0.5		50.22	1						
	315.9	0.5			1						
	327.5	0.5			1						
IL9/9	483.6	1	481.6	67.69	1	64.83	416.8	0.2	2083.9	31.8	
	489.3	1		64.33	1						
	471.9	1		62.47	1						
PT9/17	347.3	0.5	683.8	78.91	11	72.85	610.9	0.2	3054.5		
	338.2	0.5		73.67	1						
	340.1	0.5		65.96	1						
IL9/17	469.7	1	464.4	77.76	1	74.75	389.7	0.2	1948.4	36.2	
	463.8	1		74.59	1						
	459.8	1		71.89	1	·					<u></u>
Natasi									<u> </u>	<u>+</u>	
Notes:	Dilletter	5 9.									.
Run date	Dilution	File									
12-Aug	D30	ZW1 D30 F	50		<u> </u>			·····			ł
9-Sep	D20	ZW 1 D20 F	50			<u> </u>					<u> </u>
17-Sep		LANK DZU FS	00	1	1	1	1			1	1

Note: Sample ID including PT are process tank grab samples and IL are grab or composite samples from permeate as described in analysis method description.

BOD ₅ Results			Volume of	BOD bottle (mL) =>	300		
Run	Sample	Date	Day 0	Day 5	Sample	BOD ₅	Method
· · · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·				mL	mgO ₂ /L	
ZW1 D20 F50	PT	9/9/2003	7.63	3.17	1.00	1116	Titrate
ZW1 D20 F50	PT	9/9/2003	7.47	3.12	1.00	1130	Titrate
ZW1 D20 F50	PT	9/9/2003	7.72	3.04	1.00	1155	Titrate
ZW1 D20 F50	IL	9/9/2003	7.77	3.89	1.00	900	Titrate
ZW1 D20 F50	IL *	9/9/2003	7.77	4.12	1.00	831	Titrate
ZW1 D20 F50	IL *	9/9/2003	7.77	4.07	1.00	846	Titrate
	GGA	9/9/2003	7.61	4.06	6.00	141	Titrate
	GGA	9/9/2003	7.32	4.04	6.00	142	Titrate
	Blank	9/9/2003	8.15	6.85			Titrate
	Blank	9/9/2003	7.92	6.93			Titrate
······································	Seed	9/9/2003	7.71	6.49	0.50		Titrate
	Seed	9/9/2003	7.28	6.70	0.50		Titrate
				1			1
ZW2 D20 F50	PT	9/17/2003	7.00	3.53	1.00	904	Titrate
ZW2 D20 F50	PT	9/17/2003	7.05	3.39	1.00	946	Titrate
ZW2 D20 F50	PT	9/17/2003	7.25	3.30	1.00	973	Titrate
ZW2 D20 F50	IL	9/17/2003	7.35	4.38	1.00	649	Titrate
ZW2 D20 F50	IL	9/17/2003	7.35	4.26	1.00	685	Titrate
ZW2 D20 F50	IL	9/17/2003	7.29	4.73	1.00	544	Titrate
······	GGA	9/17/2003	7.33	4.12	6.00	121	Titrate
	GGA	9/17/2003	7.45	4.02	6.00	126	Titrate
	Blank	9/17/2003	7.57	6.50			Titrate
	Blank	9/17/2003	7.65	6.59			Titrate
	Seed	9/17/2003	7.73	6.39	0.50		Titrate
	Seed	9/17/2003	7.44	6.29	0.50		Titrate
						t	
* Day O - single meas	surement ava	ailable for IL					+

Table D6	5-Day Biochemical	Oxygen Demand	Analysis	(D_{20})
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composite samples from permeate as indicated in analysis method description.

TKN/ Results			[HCI]	0.005	HCI cal	libration	9.49
Method 4500-N	Retest		Blank titrant	0.000	mL	···· <u>·</u> ····	estimated
Date Completed	10/27/2003		Endpoint pH	4.896			
ID	Titrant	T-B adi.	Sample volume	Nitrogen	Mean	RSD	Comment
	mL	mL	mL	mgN/L	mgN/L	%	
ZW1D20F50	9/9/2003						
PT/10-1	6.2965	6.63	1	465	449	3.5	
PT/10-2	5.8660	6.18	1	433			
PT5-1	3.0429	3.21	0.5	449			
PT/5-1	2.5035	2.64	0.5	370			leak
IL/10-1	4.4397	4.68	1	328	344	10.7	
IL/10-2	4.0667	4.29	1	300			
IL/5-1	2.5572	2.69	0.5	377			
IL/5-2	2.5180	2.65	0.5	372			
ZW2D20F50	9/17/2003						
PT/10-1	5.7234	6.03	1	422	431	9.8	
PT/10-2	6.2746	6.61	1	463			
PT5-1	3.1435	3.31	0.5	464			
PT/5-1	2.5395	2.68	0.5	375			
IL/10-1	4.6837	4.94	1	346	349	6.8	
IL/10-2	4.4372	4.68	1	327			
IL/5-1	2.5336	2.67	0.5	374			
IL/5-2	1.1194	1.18	0.5	165			leak
Std-1	17.1432	18.06	25	51			50mgN/L
Std-2 *	7.3100	7.70	25	22			50mgN/L
* too much DI add	ded						

Table D7 Total Kjedahl Nitrogen Analysis (D₂₀)

composite samples from permeate as indicated in analysis method description.

NH3 Results		10 mil 1	[HCI]	0.005	HCI cal	ibration(9.49
Method 4500-N	Retest		Blank titrant	0.1209	mL		estimated
Date Completed	10/28/2003		Endpoint pH	4.8			
ID	Titrant	T-B adj.	Sample volume	Nitrogen	Mean	RSD	Comment
	mL	mL	mL	mgN/L	mgN/L	%	
ZW1D20F50	9/9/2003						
PT/10-1	5.1231	5.27	1	369	352	7.0	
PT/10-2	4.6406	4.76	1	334			
IL/10-1	4.9522	5.09	1	357	351	2.4	
IL/10-2	4.7954	4.93	1	345			
ZW2D20F50	9/17/2003						
PT/10-1	5.0972	5.24	1	367	370	0.9	
PT/10-2	5.1562	5.31	1	372			
IL/10-1	4.4974	4.61	1	323	326	1.3	
IL/10-2	4.5723	4.69	1	329			
Std-1	17.6470	18.47	25	52			50mgN/L
Std-2 **	17.0960	17.89	25	50			50mgN/L
Blank1	0.0938	-0.03					
Blank2	0.1480	0.03					
** leak							

Table D8 Ammonia Analysis (D₂₀)

composite samples from permeate as indicated in analysis description.

Total Phosporo	us Results		Conversion	0.0179				
Method	4500-P C							
Date Completed	9/24/2003							
ID	Abs. @ 470nm	PO₄ ⁻³ -P	PO₄ ⁻³ -P /100 mL	Sample	PO4-3-P	Average	RSD	Comments
	cm⁻¹	mg/L	mg	mL	mg/L	mg/L	%	
Blank	0.000	0.000	0.000	0.00	0.00			
Std 1	0.045	2.514	0.251	5.00	2.51	2.54	1.27	9/24/2003
Std 2	0.046	2.570	0.257	5.00	2.57			9/24/2003
Std 1	0.046	2.570	0.257	5.00	2.57			9/25/2003
Std 2	0.045	2.514	0.251	5.00	2.51			9/25/2003
ZW1D20F50								
PT9/9-1	0.053	2.961	0.296	8.00	37.0	38.4	2.57	9/24/2003
PT9/9-2	0.056	3.128	0.313	8.00	39.1			9/24/2003
PT9/9-1	0.055	3.073	0.307	8.00	38.4			9/25/2003
PT9/9-2	0.056	3.128	0.313	8.00	39.1			9/25/2003
IL9/9-1	0.029	1.620	0.162	8.00	20.3	20.3	0.00	9/24/2003
IL9/9-2	0.029	1.620	0.162	8.00	20.3			9/24/2003
IL9/9-1	0.029	1.620	0.162	8.00	20.3			9/25/2003
ZW2D20F50								
PT9/17-1	0.052	2.905	0.291	8.00	36.3	37.5	7.05	9/24/2003
PT9/17-2	0.051	2.849	0.285	8.00	35.6			9/24/2003
PT9/17-1	0.058	3.240	0.324	8.00	40.5			9/25/2003
IL9/17-1	0.025	1.397	0.140	8.00	17.5	17.9	2.25	9/24/2003
IL9/17-2	0.026	1.453	0.145	8.00	18.2			9/24/2003
IL9/17-1	0.026	1.453	0.145	8.00	18.2			9/25/2003
* Check for sulp	hide interference							
PT9/9-1 + Std	0.107	5.978	0.598	13.00	46.0	43.8		9/25/2003
PT9/9-2 + Std	0.097	5.419	0.542	13.00	41.7			9/25/2003
IL9/9-1 + Std	0.073	4.078	0.408	13.00	31.4			9/25/2003
PT9/17-1+ Std	0.102	5.698	0.570	13.00	43.8	43.8		9/25/2003
IL9/17-1 + Std	0.072	4.022	0.402	13.00	30.9	30.9		9/25/2003
	Std+Sample	Sample	Residual					
PT9/9	0.102	0.056	0.047					
IL9/9	0.073	0.029	0.044					
PT9/17	0.102	0.058	0.044					
IL9/17	0.072	0.026	0.046					
		Avg=>	0.0451	<=>	0.045	<=Avg. Std		

Table D9 Total Phosphorus Analysis (D₂₀)

composite samples from permeate as indicated in analysis method description.

Metals Analysis		ZW ₂ D ₃₀ F ₅₀						
Sample	Process	Permeate	DI Control	Redu	ction	MDL	CGDWQ	Livestock
	mg/L	mg/L	mg/L	%	mg/L		mg/L	
Al	0.566	0.038	0.018	93.3	0.53	0.001	5.0	
Ca	43.2	27.2	<0.3	37.0	16.00	0.3	1000	
Cu	0.189	0.282	0.003	-49.2	-0.09	0.0002	5.0	
Fe	2.13	0.66	0.33	69.0	1.47	0.01	0.3	AO
Mg	4.9	4.4	<0.2	10.2	0.50	0.2		
Р	71.1	62.2	<0.1	12.5	8.90	0.1		
κ	370	389	<0.3	-5.1	-19.00	0.3	300	*
Si	2.66	2.87	0.16	-7.9	-0.21	0.04		
Na	160	169	<0.5	-5.6	-9.00	0.5	300	*
S	35.5	25	<0.2	29.6	10.50	0.2		
Zn	0.451	0.0625	0.016	86.1	0.39	0.0006	50.0	
Sample #	484966	484965	484964					
Maxxam Job Number		CA317902						
Subset from Maxxam Labs			9/9/2003					
AO = Aesthetic objective								
*Depends on alkalinity and pH. Na and K should be added together when evaluating.								

Table D10 Metal Analysis of Effluents from D_{30} Run of ZW_1

APPENDIX E – MEMBRANE DESIGN CALCULATIONS FOR FULL SCALE SWINE FACILITIES

System Design Example

Using the flux values determined for the D_{20} best run, a membrane sizing calculation for a grower-finisher (feeder) pig barn equivalent to the pig production of 1000 sows was made for discussion purposes. The author's knowledge of swine production units and operation was used and supplemented by cited references commonly used in designing swine facilities.

1000 sows @ 23 pigs/sow/year => 6192 pigs in barn

Estimate of animal weight in barn

Growers (50 to 120 lbs)	1769 X 38.6 kg =	68,283 kg
Finishers (120 to 230 lbs)	4423 X 79.5 kg =	<u>351,828 kg</u>
Total		420,177 kg

Fresh Manure Production (ASAE D384.1)

$$420,177kg \bullet \frac{84kg}{1,000kg.day} \bullet \frac{m^3}{990kg} \bullet 1,000\frac{L}{m^3} = 35,651\frac{L}{day}$$

Add Dilution Water (includes waterer spillage, floor washing, dilution where required) Dilution factor = 1.4 CPS M3700 (Canada Plan Service 1989)

$$35,651 \frac{L}{day} \bullet 1.4 = 49,911 \frac{L}{day}$$
Bench Top System Data for ZW₁ and ZW₂ for D₂₀ Best Run
Average normalized flux
$$J_{20} = \frac{1.42 + 1.30}{2} = 1.36 \frac{L}{h.m^2.kPa}$$
Average Operating Pressure
$$\frac{28.7 + 25.6}{2} = 27.2kPa$$
Average COD
$$\frac{2761 + 2594}{2} = 2677.5mg/L$$

Membrane Size for 20% SWSN for Waste Produced from 6192 Feeder Pigs

To obtain 20% SWSN a continuous dilution or recycle of 80% of the permeate production would be required. An initial COD concentration of 2677.5 may be targeted but due to

the high variability of raw swine waste, monitoring and adjustments would be required Approximately 10% by volume sludge build up occurs in natural settling to produce SWSN. Use estimate of 20% by volume that would be continually wasted from system leaving 80% for membrane treatment.

$$\frac{1}{0.2} \bullet 49,911 \frac{L}{day} \bullet 0.80X \frac{day}{24h} \bullet \frac{h.m^2 kPa}{1.36L} \bullet \frac{1}{27.2kPa} = 225m^2$$

or
$$225m^2 \bullet \frac{1}{6192\,pigs} = 0.036 \frac{m^2}{pig}$$

Note: ZW-1 module has 0.047m² membrane area

Water Use Estimate

Estimated feeder pig water use including wash water (AAFRD 2000)

$$1.5Igpd \bullet \frac{4.54L}{Igpd} = 6.81 \frac{L}{day.pig}$$
$$6,192 pigs \bullet 6.81 \frac{L}{day.pig} = 42,168 \frac{L}{day}$$

1/5 of production from 225 m² membrane could supply 18.9% of water needs

or

$$(49,911\frac{L}{day} \bullet 0.80 \bullet \frac{1}{5} = 7,986\frac{L}{day} \text{ or } \frac{7,986}{42,168} \bullet 100 = 18.9\%)$$

Breeding	Feeder	Waste	Water Needs	Membrane
Sows	Pigs	Production	(L/day)	Size (m^2)
		(L/day)		
500	3,096	24,956	21,084	113
1,000	6,192	49,911	42,168	225
3,000	18,577	149,733	126,504	675
5,000	30,962	249,955	210,840	1,125
10,000	61,923	499,110	421,680	2,250