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DEVELOPMENT OF A FIELD-DEPLOYABLE MEMBRANE BIOREACTOR/SEPARATOR FOR RENDERING FACILITY WASTEWATER TREATMENT

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Lay Summary:

We built a field-deployable system for testing membrane separations in rendering plant wastewater treatment processes. This grew out of the rapid development of membrane bioreactors being deployed around the country in municipal wastewater treatment. It also builds on recent ACREC-funded work at Clemson showing that membrane processes are viable, especially for accomplishing separations without addition of polymers or other coagulants/additives that can affect the rendering plant product. The next crucial step in bringing membrane technologies into practice was long-term (several month) testing using live wastewater in continuous operation. This usually necessitates pilot tests with fairly expensive equipment and on-site operators. Our goal was to develop small systems (one pallet or smaller) that can be deployed at the rendering plant, but operated autonomously, with occasional remote control by university researchers. We have gained expertise in building autonomous and remotely controlled membrane systems at Clemson over the past several years. These "lab-on-apallet" systems will enable experiments that bridge the gap between the university lab and the real world.

Objective (s):

- 1. Create a prototype field-deployable semi-autonomous membrane separation unit (SAMSU).¹
- 2. Operate the SAMSU continuously at Clemson University for three months with no maintenance visits during the final month.
- 3. Deploy the SAMSU to a rendering facility (such as the Valley Proteins plant in Ward, SC) and test its performance during a three-month trial.

Project Overview:

Introduction -----

Membrane bioreactors (MBRs) have received a great deal of attention in recent years. The focus has been largely on municipal wastewater, but industrial treatment has also received a reasonable level of attention, particularly for high-strength wastewaters [1]. The rendering industry has another opportunity for membrane application, other than for bioreactors: membranes can be used as a primary treatment method to replace dissolved air flotation (DAF). This has the potential to reduce oxidation of fats and proteins when air is introduced in DAF. Membrane use also eliminates the need for additives like flocculant polymers that enter the rendering product stream when DAF sludge is returned to the plant [2,3].

A key difficulty with membrane processes is fouling, especially in high-strength streams and in bioreactors [4]. However, recent membrane development has resulted in very low-fouling materials [5] and when operated at low flux to prevent unnecessary buildup of foulants, stable operation can be achieved [6].

¹ After the proposal the SAMSU acronym was dropped. We simply referred to the unit as a lab-on-a-pallet membrane system.

Despite recent advances in materials and operational concepts, there remains a barrier to entry for membrane processes in the rendering industry: the membranes have only been tested in the laboratory and scale-up usually requires significant investment, with unknown return. However, membrane processes are inherently modular and can be scaled to smaller units than typical pilot plants, since each membrane sub-unit is small (such as hollow fibers) [7]. Membranes can also be automated (and usually *must* be automated because of the frequent backwash steps) and monitored remotely [8]. This makes a small pilot unit at a rendering plant feasible, even when personnel are not regularly available on-site to monitor and operate the unit.

Our research team has a history of developing automated systems and using them for membrane research [9–11]. Field deployment has not previously been our goal, but monitoring our experiments remotely (such as students checking on their laboratory experiments at night from their apartments) is routine. This experience gives us the background in membrane processes, instrumentation, sensors, control algorithms, and remote communication, which will be required to accomplish the objectives of this project.

The advantages of membrane technology lead to extensive focus and application of microfiltration (MF) in removing macromolecular pollutants [12,13]. Nowadays, at least 50 individual membrane bioreactor (MBR) manufacturers are supplying membrane modules to hundreds of large-scale MBR plants worldwide and the application of MBR systems are increasing continuously due to their limited footprint and a requirement of high effluent water quality [14]. During the past decades, ceramic membranes have received a great deal of attention in research and development due to their high chemical resistance, hydrophilic surface, thermal stability and endurance in high flux backwash [15,16]. These advantages make ceramic membranes appropriate for the treatment of high strength industrial wastewater, which usually presents high chemical oxygen demand (COD) and turbidity.

High strength industrial wastewater is defined as that wastewater with high concentrations of COD, ammonia, suspended solids, and heavy metals. It contains fats, oil and other organic or inorganic pollutants, and the exact nature of the wastewater varies with the type of industry [1]. Wastewater from the rendering industry is a kind of high strength industrial wastewater which contains significant amounts of suspended solids, fats, oils, greases and proteins [2]. Dissolved air flotation (DAF) is the most popular method for primary treatment in a rendering plant, but it has limitations. During DAF, chemicals must be added to maintain pH and enhance flocculation efficiency. Chemical addition is not desirable since the fats and proteins recovered during DAF are valuable products that are sent to the head of the facility for recovery. Polymer coagulants such as polyacrylate are contaminants in the final protein and fat products, which means their use must be carefully monitored and controlled. Also, fats and proteins have the potential to be reduced when aeration is introduced in DAF [2]. These limitations lead to this project, using ceramic membrane as a primary treatment method to replace DAF.

Fouling is a key difficulty during filtration, especially in high strength industrial wastewater [17]. Sumihar studied different cleaning strategies for cleaning ceramic membranes fouled by produced water. Temperature, cleaning pressure, chemical agents, and chemical concentration were compared [18]. Various factors like cleaning time and crossflow velocity were studied by Hongjoo for recovering the flux of ultrafiltration (UF) membranes fouled by natural organic matter (NOM) [19]. Enzymes were employed by Chen for cleaning membranes fouled by protein mixtures [20].

Although the literature cited above gives insights into membrane fouling from different areas, there remains little published data for the treatment of high strength wastewater especially of rendering plant wastewater. This project focuses on the membrane cleaning strategies for the recovery of ceramic membranes fouled by rendering wastewater. Further, the wastewater composition at a rendering plant varies by the hour and by the day; studying the effects of that variability is difficult. The system used in this research is a field-deployable membrane unit for studying the treatment of rendering effluents in the plant. Various backwash strategies and chemical agents are employed along with physical and chemical cleanings. The backwash strategies on determining the characteristics of rendering wastewater via observing different cleaning efficiencies.

This project builds on the rapid development and the natural characteristics of membranes, that frequent operation like backwash and chemical cleaning are required. Also, the water quality greatly influences the cleaning strategy.

Materials & Methods -----

We custom built our lab-on-a-pallet membrane separation unit, which was our first main objective. Components included a pH sensor, a conductivity sensor, polycarbonate sheeting for housing electrical components and for framing, tubing, AC/DC converters to power transmitters and other electrical components, a power cable, circuit breaker, terminal blocks, low-amperage shielded cable, DIN rails and electrical connectors for powering components and delivering signals to the control computer, serial to USB converter box for interfacing the computer with the balance and pumps, solenoid valves for controlling water flow during production and backwash cycles, and T-slotted structural framing material (so called "80/20" extruded aluminum) and associated brackets, hinges, and connectors for creating the frame.

Ceramic membranes were fabricated by Inopor with mean pore sizes of 100 and 200 nm. The effective membrane area is 0.025 m². The membrane module was a 316 stainless steel cell. Three peristaltic pumps were used; one for production, one for backwash, and one for cleaning. Four solenoid valves were employed to control the flow entering or leaving the module during the various steps. An actuator valve was used at the concentrate port to control pressure and flux. Two pressure gauges and transducers were used to measure the pressure at the concentrate port and backwash port. A flowmeter was installed at the permeate port to measure the pressure, temperature, and flowrate of the permeate. Three sensors were inserted into the feed tank, and pH, temperature, and conductivity were measured and recorded. A balance was mounted before the permeate tank. According to the balance reading, the flow rate was calculated, which works as a reference to the permeate flow meter. A personal computer was used for data acquisition, and several signal converter boxes were used to interface between devices and the control computer.

We also developed control algorithms (which is the second part of Objective 1). Control is provided using National Instruments Labview software, for which Clemson maintains a site license. We built a set of "virtual instruments" (VIs; this is National Instruments' terminology) based on similar VIs we have used in other membrane systems. The Vis were tailored for the specific components of the new system. The VIs were tweaked throughout the project; development of this software is one of the key activities and outcomes of the work. The programming effort was significant because the unit was intended to operate continuously and adjust itself to maintain set points even with changes in feed water quality, such as particle accumulation or pressure variations. After all the components were installed and the system was running, the entire set of VIs were rewritten to improve their efficiency and eliminate extraneous code that had accumulated during the development process.

The system can be divided into two parts: software interface and fluid-handling hardware. Figure 1 presents a schematic drawing of the membrane system. Filtration, backwash, and chemical cleaning were realized using different operation methods, shown in Figure 1.



Figure 1. Schematic of the system.

The Labview data acquisition and control system involves three main components: filtration loop control, actuator auto adjustment, and data acquisition and presentation.

The Filtration Loop Control program (Figure 2) is the fundamental VI, which adjusts pumps, solenoid valves, and the actuator valve to achieve automatic backwash and chemical cleaning after filtration. Filtration, backwash and chemical cleaning periods can be set by the operator at the beginning of a run.



Figure 2. Screenshot of the Filtration Loop Control VI.

The Filtration Loop Control VI contains a case structure and a time-based Step Choose program. The case structure has eight different cases which correspond to eight steps during filtration; they are as follows.

Step One – Filtration Prepare (FP). In this step, a sequence structure was built, which contains four settings in sequence. The first one restores all components to initial state and waits for 1 second to make sure all signals are sent to hardware. The second sets actuator input voltage at 1.5 V and waits for ten seconds, which provides enough time for the actuator valve to respond. The third one sets 1-6 solenoid valves as closed and waits for 1 second. The last one sets the backwash pump and chemical pump speed at 0 revolutions per minute (rpm) and the feed pump speed at 100 rpm, then waits for 48 seconds. This step takes 60 seconds for all of the processes for filtration prepare.

Step Two – Filtration (F). This adjusts the actuator valve automatically for maintaining transmembrane pressure (TMP) or flux close to the target TMP or flux.

Step Three – Backwash Prepare (BWP). This step is similar to Step One which also has four settings in sequence. The first one restores all components to their initial state and waits for one second to make sure all signals are sent to hardware. The second sets actuator input voltage at five V and waits for ten seconds, which provides enough time for the actuator valve to respond. The third one sets 1-6 solenoid valves as closed or open (as needed), and then waits for 1 second. The last one sets the chemical (cleaning) pump speed at 0 rpm, the feed pump speed at 100 rpm, and the backwash pump speed at 90 rpm, and then waits for 48 seconds. This step takes 60 second for all of the processes for backwash prepare.

Step Four – Backwash (BW). This step adjusts backwash pump speed automatically for maintaining backwash pressure close to target backwash pressure.

Step Five – Chemical Cleaning Prepare (ChemP). This step is similar to Step Three which also has four setting in sequence. The first and second settings is the same as Step Three. The third one sets 1-6 solenoid valves as closed and xxx as open, and then waits for 1 second. The last one sets chemical pump speed as 20 rmp and feed pump and backwash pump speed as 0 rmp; and then wait for 48 seconds. This step will cost 60 second for all of the processes for chemical cleaning prepare.

Step Six – Chemical Cleaning (Chem). This adjusts the backwash pump speed automatically for maintaining backwash pressure close to target backwash pressure.

Step Seven – Backwash after Chemical Cleaning Prepare (BWCP). This step is similar to Step Three which also has four settings in sequence. The first and second settings are the same as Step Three. The third one sets 1-6 solenoid valves as closed or open, as needed, and then waits for one second. The last one sets chemical pump speed at 0 rpm, feed pump speed at 20 rpm, and backwash pump speed at 90 rpm, and then waits for 48 seconds. This step will take 60 seconds for all of the processes for backwash after chemical cleaning prepare.

Step Eight – Backwash after Chemical Cleaning (BWC). This adjusts backwash pump speed automatically for maintaining backwash pressure, and records data.

The case structure is controlled by a Time-based Step Choose VI (Figure 3). This program aims to decide which case should run during filtration based on relative time. The prepare steps (filtration prepare step, backwash prepare step, chemical cleaning prepare step and backwash prepare after chemical cleaning prepare step) only require 60 seconds. Because of that we always set the time at 60 seconds for each prepare step in the front panel (Figure 3 shows the time setting part of the front panel at the top of the figure). For F, BW, Chem and BWC, we can set different times. Although there are eight steps during filtration, we can describe the filtration as four actions by adding each prepare step to the main step like adding FP and F as filtration, BWP and BW as backwash, ChemP and Chem as chemical cleaning, and BWCP and BWC as backwash after chemical cleaning.



Figure 3. Time-based Step Choose VI.

We set the frequency of the chemical cleaning by setting "Chem after N cycles". This setting means that after a certain number of filtration and backwash cycles there will be a chemical cleaning. For example, if we set "Chem after N cycles" as two then, the process will be: $[FP \rightarrow F \rightarrow BWP \rightarrow BW] \rightarrow [FP \rightarrow F \rightarrow BWP \rightarrow BW] \rightarrow ChemP \rightarrow Chem \rightarrow BWCP \rightarrow BWC$, which means chemical cleaning will be carried out after two groups of filtration and backwash.

The flexibility of the programs is a key feature of the system. Sometimes there is no chemical cleaning required, which means only FP, F, BWP, and BW steps are employed. We designed the Time-based Step Choose program to allow no chemical cleaning by simply setting ChemP, Chem, BWCP, and BWC to zero. On the other hand, we can also set filtration and backwash times as zero if we only want to employ chemical cleaning and backwash after chemical cleaning. Another method for canceling the chemical cleaning is setting "Chem after N cycle" as a large value (e.g. one million); the chemical cleaning would only happen after a million cycles of filtration and backwash, which would never (practically) be reached.

During filtration, the membrane is fouled by wastewater, which increases the resistance of the membrane. In this case, the TMP will increase or the Flux will decrease. An Actuator Auto Adjustment program was coded to maintain either constant pressure or constant flux. For example, in constant-flux mode the program automatically closes the valve a little to provide a higher trans-membrane pressure and maintain the same flux when the fouling increases. The Actuator Auto Adjustment VI originated from a similar program called "P Check," which was created in our previous work [21]. Figure 4 shows the block diagram of the Actuator Auto Adjustment program. The program uses case structures to compare the real TMP and the target

TMP. If the real TMP is in the range of target TMP \pm increment TMP the output signal to the actuator valve will be zero, which means no adjustment is required. If the real TMP is lower or higher than the range, the output voltage will decrease or increase after the calibration.

A calibration curve was generated in which \triangle TMP was the difference between real TMP and target TMP and \triangle V was found, then added to the old actuator voltage. There were four linear relationships in the curve. When the absolute value of \triangle TMP is smaller than 3 the slopes are much higher than the slopes at the absolute value of \triangle TMP higher than 3. Because we found that the TMP always over adjusted after the first time reaching to target TMP, we use two slops to describe the relationship between TMP and voltage in each quadrant. The third quadrant has lower slopes than the first quadrant. The reason for this setting is to avoid sinusoidal oscillation. A situation happened before this setting that the \triangle TMP was always higher than the increment, which meant the Actuator Auto Adjustment program adjusted the actuator valve continuously and the actuator voltage showed as a sine curve. We broke the sine oscillation by using lower slopes in the third quadrant, which means the \triangle V has a higher increase speed and a lower decrease speed.



Figure 4. Actuator Auto Adjustment VI.

This system was designed to be used as a test system for treatment of different wastewater with various membranes, which means the target flux or pressure should be auto-adjustable. The Auto Pressure/Flux programs were built to accommodate this need. The programs adjust the target pressure or flux according to the filtration-backwash cycle. Thus, with the actuator valve responding program, the treatment efficiency can be tested automatically.

The Data Acquisition and Presentation VI is shown in Figure 5; twenty-one data are written to a text file every ten seconds and the file is sent via email to students and professors every 3 hours.



Figure 5. Data Acquisition and Presentation VI.

We planned to deploy the unit to a rendering plant, where a side-stream of wastewater from the process was to be plumbed to the membrane system. Part of the permeate was to be stored and used for backwash, while the rest of the permeate and concentrate would go back to the plant's treatment process. Samples of permeate were to be used to measure COD to understand filtration efficiency. Unfortunately, we were unable to realize deployment to the treatment plant because the plant staff had other events arise and were unable to devote time to the project. Instead of deployment, we brought a wastewater sample to the lab and tested the system as if it were deployed remotely, by interacting with it only over the Internet for a designated length of time (several days).

Background values of wastewater like temperature, conductivity, and pH were evaluated to understand how these variables influenced the membrane performance. Basic parameters like the length of filtration time and backwash time, pump speed, and actuator voltage input were recorded to know the operation state. The pressure of concentrate and permeate, flowrate crossing membrane, and balance reading were analyzed to evaluate transmembrane pressure, flux, and resistances. These parameters were used to not only describe the membrane system, but also gave insight into the characteristics of the high strength industrial wastewater.

An online fouling monitoring method [22] was considered to determine its usefulness in evaluating fouling rate (FR), during experiments. There are three main steps of this method: (1) Concentrate pressure (P_C) and permeate pressure (P_P) are collected during filtration and transmembrane pressure is calculated automatically with Equation 1. (2) The FR is calculated

every ten minutes according to Equation 2. (3) FR values are plotted with time, and the slopes of FR versus time are used to describe wastewater characteristics in a certain target flux or pressure.

$$\mathbf{TMP} = \mathbf{P}_{\mathbf{C}} - \mathbf{P}_{\mathbf{p}} \tag{Eq. 1}$$

$$\mathbf{FR} = \frac{\Delta \mathbf{TMF}}{\Delta t}$$
(Eq. 2)

In order to evaluate the removal efficiency of the membrane, the rejection coefficient (R_C) will be calculated using Equation 3, where C_P and C_F are concentrations of permeate and feed, respectively.

$$R_C(\%) = (1 - \frac{C_P}{C_P}) * 100$$
 (Eq. 3)

The membrane resistance will be determined using the resistances-in-series model (Equation 4).

$$J_p = \frac{\Delta P}{\mu R_T} \tag{Eq. 4}$$

 J_P is the permeate flux, R_T is the total resistance against solvent permeation, and μ is the viscosity. In this model, the total membrane resistance is the sum of the intrinsic membrane resistance, R_M , and the extrinsic resistance, R_{FC} which is caused by fouling and concentration polarization. The extrinsic resistance consists of reversible resistances (R_{REV}) and irreversible resistances (R_{IRR}). Reversible resistances are caused by concentration polarization and cake layer deposition which can be easily removed by backwash. The irreversible resistances are mainly adsorption and pore blocking, which can only be removed by chemical cleaning. Each resistance can be evaluated as follows:

- R_M will be calculated according to Eq. 4, with DDI water as the feed flow.
- R_T was determined by Eq. 4 with real-time TMP, J_P , and viscosity which was calibrated with temperature.
- R_{FC} was the difference of R_T and R_M .
- R_{REV} and R_{IRR} were obtained by the VITO Fouling Measurement [23] with real-time data.

Results & Discussion ------

An overview photograph of the current lab-on-a-pallet system is shown in Figure 6. The computer and electrical components are mainly housed on the upper level, with most of the liquid-processing components on the lower level, which reduced the chance for spills causing electrical shorts. Though "pallet" is the size term we have used, the actual design was made narrow enough to fit through typical doorways.



Figure 6. Lab-on-a-pallet membrane system.

Some key liquid components on the lower level can be seen in Figure 7. These components were reconfigured to fit the new frame. The membrane cell is designed for ceramic membranes, but can be easily switched in the future for testing other membrane types. Three solenoid valves (black components connected to tubes) and one actuator-driven needle valve (red box near rear) control water flow.

Most electrical components were centralized in a panel at the front of the unit for easy access during development and operation (Figure 8). The panel was made with polycarbonate sheeting so that users can observe sensor displays and lights on the various components. A major effort was wiring the data acquisition and control modules correctly with their sensors, and connecting these to the computer. For example, the white box just above and left of center in Figure 8 is the analog input card that reads pressure transducer, conductivity, and pH signals (4-20 mA) and outputs valve actuator control signals (0-10 V). The gray box to the right just below center is the

solenoid valve switch box. The white box on the lower right is the serial connection module that interfaces with the balance, the feed pump, and the backwash pump. These three boxes connect to the computer through USB interfaces. The blue-green and blue displays on the left are the pH and conductivity displays (respectively). The three blue, vertically-aligned boxes near the top are the A/C to DC power supplies for the sensors. Other visible components are wiring conduits and terminal blocks.



Figure 7. Key components of fluid handling, including feed and backwash pump and ceramic membrane module.



Figure 8. Electrical panel of the lab-on-a-pallet system showing data acquisition and control components.

Backwash is an important part of continuous membrane operation. In lab-scale membrane research it is rare that investigators include backwash capabilities in their experimental designs because this is seen as more of a scale-up issue and because it increases the complexity of the test setup; however, for a thorough evaluation of membrane performance, backwash is needed. This is especially true for high-strength industrial wastewater where blinding can occur quickly and a backwash pulse is needed to maintain membrane operability.

Even with backwash, membrane performance decreases over time and cleaning is required. In a laboratory setting this would be done manually, but for a remotely operated system online cleaning is useful. Further, there is a recent hypothesis emerging in the membrane community about "critical flux" related to cleaning: if a membrane is operated below its critical flux, then cleaning will completely restore the original flux and long-term operation can be sustained. The critical flux idea has been explored related to backwash, but has not been thoroughly explored with respect to cleaning. We now have a system that can test this hypothesis.



Figure 9. Pressure, flux, and temperature data from a one-day filtration experiment.

With backwash and cleaning capabilities in place, the system was ready for programing test protocols. The end-goal is for the user to press "go" on a test sequence and the system will automatically perform the desired experiment. One such experiment is related to the critical flux; the pressure is increased at regular intervals over a defined time period, then systematically decreased in a similar manner. Data were recorded and analyzed (Figure 9) to evaluate performance. As currently designed, the system records 21 parameters including pressure, permeate flow rate, temperature, pH, conductivity, backwash pressure, and valve actuator set

point. This enables a thorough analysis of system conditions and will enable modeling of the system to predict performance after a sufficient set of training data are collected.

The lab-on-a-pallet system was able to operate for long time periods (several hours to several days) to thoroughly evaluate membrane performance with backwash and cleaning cycles. An example data set (Figure 3) that used raw rendering plant wastewater as feed shows that relatively stable cyclical operation was achieved. Much of the fouling was reversible via short backwash applications, which are visible in the rebound after each short (~23 minute) filtration period. Cleaning (at 2.7 and 5.3 hours) with NaOH was successful in further removing foulants and restoring flux. However, flux during each filtration period did rapidly decline as fats, proteins, and other organic matter attached to the aluminum oxide membrane. It is anticipated that surface-modified membranes developed in future work will have less rapid flux decline during each filtration cycle, enabling longer periods between backwash. This will increase the overall water recovery and reduce cleaning costs. It will also mean that new membrane installations will require less membrane area, decreasing capital costs.



Figure 10. Eight hours of permeate flow rate data for a ceramic membrane in the lab-on-a-pallet system. Backwash was performed at roughly 23 minute intervals. Cleaning with NaOH was performed at 2.7 and 5.3 hours.

Conclusions -----

The lab-on-a-pallet membrane system was successfully built and operated remotely. We were not able to deploy the unit to a rendering plant because of problems that arose with our partner facility; however, we achieved the goal of testing rendering plant wastewater and doing so with only control via Internet for at least six days. References -----

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Impacts and Significance:

This project successfully developed a platform for testing membrane processes at rendering facilities. Further, we showed that ceramic membranes can be operated for long time periods with regular backwash and cleaning cycles. We anticipate that future membrane development will further improve performance.

Publications:

Ladner, D.A. "Exploring and optimizing membrane treatment facilities." Platform presentation at the Technology Transfer Conference, Greenville, SC (January, 2017).

Ladner, D.A. "Membrane processes in drinking water treatment: MF to RO and PVDF to polyamide." Platform presentation at the Technology Transfer Conference, Greenville, SC (January, 2017).

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Outside funding:

Funded project: "Anaerobic Membrane Bioreactors as a Next-Generation Technology to Address the Food-Energy-Water Nexus." State of South Carolina EPSCoR Stimulus Research Program, March 2018.

Funded project: "Targetting NSF INFEWS/T3 Funding and Promoting Indurstry-Academia Collaboration for Next-Generation Resource Recovery from Wastewater." TIGER Grant, Clemson University College of Engineering, Computing, and Applied Sciences, January 2018.

Proposal submitted: "INFEWS/T3: Anaerobic Membrane Bioreactor as Next-Generation Municipal Wastewater Treatment for Resource Recovery: Potable Water, Energy, and Nutrients." National Science Foundation, Innovations at the Nexus of Food Energy and Water (INFEWS) program, March 2017. Was not funded, but received several favorable reviews.

Future Work:

We are applying for ACREC funding to extend this work to anaerobic membrane bioreactors (AnMBRs). We are also planning to submit a large-scale proposal to the National Science Foundation INFEWS program.

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