[2014] Project Abstract For the Period Ending June 30, 2017

PROJECT TITLE: Next Generation Large-Scale Septic Tank Systems PROJECT MANAGER: Bo Hu AFFILIATION: University of Minnesota MAILING ADDRESS: 1390 Eckles Ave CITY/STATE/ZIP: St Paul, MN, 55108 PHONE: (612) 625-4215 E-MAIL: bhu@umn.edu WEBSITE: http://bohu.cfans.umn.edu/ FUNDING SOURCE: Environment and Natural Resources Trust Fund LEGAL CITATION: M.L. 2014, Chp. 226, Sec. 2, Subd. 08g

APPROPRIATION AMOUNT: \$258,000 AMOUNT SPENT: \$258,000 AMOUNT REMAINING: \$0

Overall Project Outcomes and Results

Septic systems treat sewage generated from homes and mid-sized facilities that do not have access to centralized wastewater treatment plants. They are effective in retaining and degrading organic matters in the sewage, however, the tanks do not substantially decrease phosphorus nutrients in effluent. The nutrients may enter surrounding aquatic environment and have a negative impact in water bodies such as lakes and rivers. To overcome the limitation, we developed a novel septic system that is better at removing phosphorus and organic matter than conventional septic tanks by using a "microbial electrolytic cell" which uses electricity to promote biological reactions inside the tank. Different reactor designs, sizes and operational parameters were investigated, i.e., from laboratory scale to pilot scale. Laboratory scale experiments were carried out at the University of Minnesota - Department of Bioproducts and Biosystems Engineering and the pilot scale was tested at the Saint Paul Municipal Wastewater Treatment Plant of Metropolitan Council Environmental Services. A preliminary technoeconomic assessment showed that around one third additional installation cost is needed to implement this technology, and this enables 50%-90% of the phosphorus removal from the sewage, compared to the conventional septic system. The collaboration with the Metropolitan Council Environmental Services led to the development of a new technology that can help communities effectively manage their wastewater treatment systems and improve public health and the environment. The technology developed from this project, together with the information obtained from the techno-economic analysis, could be useful to thousands of rural communities and help decision-making process of trying the novel tank configuration. When communities effectively manage their wastewater treatment systems, public health and the environment are adequately protected while the community has the management structure in place over the long-term.

Project Results Use and Dissemination

The technology developed from this project, together with the information obtained from the technoeconomic analysis, can be beneficial to Minnesota septic tank users, which account for about 20%-25% of its population. Besides the academic dissemination, a video of showcasing the pilot-scale testing system was posted in our website (http://bohu.cfans.umn.edu/) and youtube (https://z.umn.edu/lccmrvideo) for general public access. Important and practical results from this project will also be disseminated in relevant workshops or in Onsite Sewage Treatment Program of UMN. During the study, project information, results, and major achievements have been disseminated through multiple ways. While we are preparing another two manuscripts for peer-reviewed publication, one paper was published. Four oral presentations were made in 2014 and 2016 AIChE conferences, and in 2015 and 2016 ASABE AIM conferences. Two posters were also presented in the Department of Bioproducts and Biosystems Engineering Showcase.



Environment and Natural Resources Trust Fund (ENRTF) M.L. 2014 Work Plan Final Report

| Date of Report: | August 11, 2017 | | | |
|---|-----------------|--|--|--|
| Final Report | | | | |
| Date of Work Plan Approval: | June 4, 2014 | | | |
| Project Completion Date: | June 30, 2017 | | | |
| Does this submission include an amendment request? No | | | | |

PROJECT TITLE: Next Generation Large-Scale Septic Tank Systems

| Project Manager: | Bo Hu |
|----------------------|--|
| Organization: | University of Minnesota |
| Mailing Address: | 1390 Eckles Ave |
| City/State/Zip Code: | St Paul, MN, 55108 |
| Telephone Number: | (612) 625-4215 |
| Email Address: | bhu@umn.edu |
| Web Address: | http://www.bbe.umn.edu/People/Hu/index.htm |

Location: The experiment will be primarily done at Biological Agricultural Engineering Building (BAE) 320, 1390 Eckles Ave, St Paul, MN, 55108. The impact of the project will be statewide

| Total ENRTF Project Budget: | ENRTF Appropriation: | \$258,000 |
|-----------------------------|----------------------|-----------|
| | Amount Spent: | \$258,000 |
| | Balance: | \$0 |

Legal Citation: M.L. 2014, Chp. 226, Sec. 2, Subd. 08g

Appropriation Language:

\$258,000 the second year is from the trust fund to the Board of Regents of the University of Minnesota to develop a dual utility large-scale septic tank system designed for nutrient recuperation, bioenergy generation, and environmental protection using a bio-electrochemical system. This appropriation is subject to Minnesota Statutes, section 116P.10. This appropriation is available until June 30, 2017, by which time the project must be completed and final products delivered.

I. PROJECT TITLE: Next Generation Large-Scale Septic Tank Systems

II. PROJECT STATEMENT:

Subsurface Sewage Treatment Systems, commonly known as septic tank systems, aim to treat sewage generated from homes and mid-sized facilities that do not have access to centralized wastewater treatment plants. Nearly 25% of the US population is served by an onsite wastewater treatment system as their primary means of wastewater treatment. The primary wastewater treatment of conventional septic tanks is limited since the system relies on the capacity of retaining suspended solids by accumulation and sedimentation. Furthermore, most of the dissolved organics (soluble organic matter) and nutrients (nitrogen and phosphorous) need further soil treatment and can cause environmental problems such as eutrophication in water bodies if not properly treated. The waste stream in the septic tanks is a great potential source to recover phosphorus. Septic tanks also emit instead of collect powerful GHGs, such as methane (CH₄) and nitrous oxide (N₂O) to the atmosphere, as well as hydrogen sulfide (H₂S), which causes obnoxious odors and concrete corrosion. The role of anaerobic digestion (AD) for closing the water and the nutrient cycle has been considered of importance during onsite wastewater treatment. AD has the capability of generating energy from organic matter as biogas while decreasing the amount of pathogens. However, AD has only a limited capability to remove other pollutants such as nitrogen (N) and phosphorus (P).

Electrochemical assisted AD has been recently introduced as a new alternative to improve the AD process and to allow nutrient recuperation. The implementation of a bio-electrochemical system such as a microbial electrolysis cell (MEC) coupled with a digester can allow *in situ* production of hydrogen (H₂), oxygen (O₂) and precipitation of phosphorus salts such as struvite (MgNH₄PO₄·6H₂O) by applying an electric field. Therefore, an increase of the energy content of the biogas can be expected due to a higher H₂ concentration or direct methanogenesis by converting electrons, protons and CO₂ into CH₄. A decreased H₂S concentration in the biogas can be achieved by the micro-aeration effect. Meanwhile, phosphorous concentration in effluent can also be lowered by salt precipitation at electrodes.

This project aims to develop next generation septic systems focusing on nutrient recuperation, bioenergy generation and environmental protection by the implementation of a bio-electrochemical system. This project proposes to plug a microbial electrolysis cell (MEC) into current septic tank systems in order to improve the water quality of septic tanks effluents, to recuperate phosphorus that can be used as fertilizer, to increase the production and collection of biogas for the bioenergy application and to decrease the greenhouse gas (GHGs) emissions. The experimentation will be carried out at the lab and at the field, and the results obtained will be applied to modify current design of the septic tank systems. The project will evaluate the capital and operational costs of the implementation of such a system and assess the potential benefits. The technology developed during this project could be useful to thousands of rural communities, especially those that do not have access to centralized wastewater treatment facilities. When communities effectively manage their wastewater treatment systems, public health and the environment are adequately protected while the community has the management structure in place to sustainably treat their wastewater over the long-term.

III. PROJECT STATUS UPDATES:

Amendment Request 06/30/2017:

We request to transfer \$2,621 from Personal (Wages and Benefits), \$1,025 from publication cost, and \$2,692 from travel expenses to Equipment/Tools/Supplies to cover the lab supplies and analysis service. We also request to transfer \$270 from travel expenses to capital expenditures to build a MEC reactor. There are some minor deficits and surplus over the three project years and this is the final rebudget request to allocate some funds to match the deficit and surplus. We are short of supplies and lab services while the funds for personal, travel and publications have small surplus.

Amendment Request 01/01/2016:

We request to transfer \$10,000 from Personal (Wages and Benefits) to Equipment/Tools/Supplies to cover the lab supplies and analysis service. The postdoc researcher joined the project team a few months late and did not consume the funding as we originally budgeted; however, the sample analysis and lab supplies cost more than what we originally thought. Therefore, we are requesting to decrease the funding for Personal for the first activity from \$109,205 to \$99,205 and increase the budget for Equipment/Tools/Supplies from \$4,575 to \$14,575 for the first activity. The rest of the budget remains same.

Amendment approved by the LCCMR January 20, 2016

Amendment Request 08/18/2014:

We want to transfer \$5,000 non-capital equipment budget and \$3,000 for lab supplies to capital equipment so that we can use it to contribute a detector purchase for a HPLC. This \$5,000 was originally budgeted to set up two lab scale CSTR reactors. We have already built these two reactors in the lab and start to prepare for the chemical analysis for the samples generated from these reactors. However, our sample analysis is limited to our current capability. We are in the process to purchase a HPLC and this will facilitate our sample analysis for the project. With this HPLC, we are expecting that the cost for lab supplies related to sample analysis will be decreased also, because it is very expensive to buy the test kits and with the HPLC we do not need to have some specific test kits anymore. The HPLC cost is \$31,496.88 and we request to transfer this \$5,000 for non-capital equipment and \$3,000 for lab supplies to contribute to this HPLC purchase. The rest of the funds for this HPLC purchase will come from the research group.

Amendment approved by the LCCMR August 20, 2014

Project Status as of January 1, 2015:

The Next Generation Septic Tank project is making good progress. Postdoctoral researcher has been appointed for conducting experiments and writing reports. Besides the regular weekly group meeting to update the project, two brainstorming project meetings were held to discuss the project. According to the work plan, batch and continuous reactors were constructed (Outcome 1, Activity 1). Electrode evaluation for redox activity and internal resistance for electrochemical cells in different media composition were carried out already. Anaerobic digestion at different temperatures, electrode combinations, and external voltages were conducted for evaluate the biogas production and nutrient removal from supernatant liquid. As an extension of the project, some sacrificing electrodes (Fe and Al) were tested for effects on phosphorus and solids removal. Future efforts (Outcome 2, Activity 1) will be focused on microbial anode development and analysis based on electrode materials of carbon cloth, stainless steel mesh, carbon iron and cast iron plate, and mixed metal oxides.

Project Status as of July 1, 2015:

We are continuing making process on the Next Generation Septic Tank project. Regular weekly group meeting was help to update the project, and the postdoc researcher gave a presentation once per month to my whole research group to explain the detailed research results and plan for the next month. Two additional brainstorming project meetings were held with everybody in the project to discuss the future project goals. According to the work plan, lab-scale prototype systems were studied, primarily focusing on evaluating different electrode materials. We have made the conclusion to select SS 430 as the most suitable electrode material. We further studied different lab scale reaction operations to understand mechanisms for the increased biogas production and phosphorus crystallization in order to finalize the best reactor design (Outcome 2). Continuous reactor operations are currently underway in order to study the possible biofilm growth on anode and evaluate the effect of biofilm on phosphorus removal.

Project Status as of January 1, 2016:

The progress of the project continues according to schedule. Dr. Carlos Zamalloa has joined the project. In accordance to the Activity 1 – Lab-scale reactor optimization, two continue stirred tank reactors (CSTR's) are being

operated in order to find optimal operational parameters as part of the preliminary assessment. The two CSTR's ('Control' reactor and 'CSTR-MEC' reactor) were prepared with a working volume of 1.6 L. Both reactors are operated in continuous mode under mesophilic conditions (35°C ±2°C) at a hydraulic retention time of 32 ±0.2 days and at organic loading rates (OLR) of 0.9 ±0.04 gCOD/L.day. Furthermore, both reactors contain an anode projected area of 308 cm² and a cathode projected area of 375 cm². The Control reactor is operated in open circuit mode and the CSTR-MEC reactor is operated with an applied voltage (Eap) of 0.52 ±0.01 Volts. Under these conditions, the total COD recovered as biogas was in the same order of magnitude in both reactors i.e. 41.5 ±2.6% for the Control reactor and 43.0% ±2.6% for the CSTR-MEC reactor. Moreover, the methane concentration in the biogas is similar in both reactors i.e. about 61%. Yet, the concentration of hydrogen sulfide (H₂S) was about 50% lower in the CSTR-MEC reactor. Interestingly, the amount of total ammonium nitrogen (TAN) and of total phosphorous in the effluents of the CSTR-MEC reactor are about 20% lower than for the Control reactor indicating that struvite (NH₄MgPO₄) might be precipitating in the reactor. This preliminary work indicates that the CSTR-MEC is capable of removing H₂S from the biogas and removing phosphorous and nitrogen from the effluent. In the following months, other operational parameters such as OLR, HRT and Eap will be studied. In addition, mass balances will be carried out to estimate the removal efficiencies of the system. At the end of the operation of the reactors, linear sweep voltammetry (LSV) and cyclic voltammetry (CV) will be performed to collect valuable information on the electron transfer mechanisms in the reactors. In addition, a lab-scale prototype with high projected electrode area per unit of reactor is being build.

In addition, 14 reactors with a working volume of 1 L were prepared and operated at 20°C ±2°C and 28°C ±2°C. Our results indicated that voltage levels larger than 0.5 V is necessary to achieve sufficient P removal which may work for sewage run through septic tanks with normal HRTs (e.g., 3-5 days), but levels smaller than 1.0 V is required to avoid solids buildup. Therefore, four levels of voltages (0.50 V, 0.63 V, 0.75 V, and 0.88 V) were applied to reactors. Raw sewage without primary settling was obtained from Blue Lake Wastewater Treatment Plant, Shakopee, MN, as influent for the reactors. Before being used as reactor feed, sewage was sieved by 50 mesh to remove big particles. 500 mL of reactor medium was replaced by sewage at every feeding cycle at an interval of 3, 4, or 5 days. An average HRT of 8.3 day (feeding cycle of 4.17 days) was used for the 171 days' operation. Under these conditions, phosphorus was slightly removed in the control reactors, 12.2% at 25 °C or 7.45% at 15 °C, possibly by sedimentation of particles to the sludge (or sediment) layer of the reactors. The voltage application substantially increased phosphorus removal, to between 77.18% and 98.65% at 0.50 V to 0.88 V at 25 °C, and to between 20.7% and 89.5% at 0.50 V to 0.88 V at 15 °C. TP levels were dropped to 0.11 mg/L or 0.49 mg/L at the best scenarios. However, fluctuations in effluent P levels were observed in some reactors, especially at lower temperature, the cause of which was not clear at this point. When voltage increased to 0.88 V, effluent TP levels became more stable. Figure 23 shows the TP evolution in one feeding cycle, which suggested that most TP removal occurred in the first 24 h and therefore it can be practical to further reduce HRT with TP removal performance maintained. Figure 24 shows that almost all phosphorus in effluent samples was in the form of reactive (PO_4^{3-} , HPO_4^{2-} , and $H_2PO_4^{-}$), while in influent sewage reactive P accounted for 87%.

Project Status as of July 1, 2016:

Continuing previous activities, two continue stirred tank reactors (CSTR's) termed 'Control' reactor and 'CSTR-MEC' reactor with a working volume of 1.6 L were operated in continuous mode under mesophilic conditions $(35^{\circ}C \pm 2^{\circ}C)$ for a period of 160 days in four operational phases (Table 7). During the first three phases, the HRT was decreased from about 30 to 25 days in Phase B to 20 days in Phase C and D. As expected, the amount of biogas produced by each reactor increased due to the increase of the organic loading however clear differences between both reactors in terms of biogas production, biogas composition and conversions efficiency were not observed. The conversion efficiencies during phase A to C were in the same order of magnitude for both reactors i.e. about 40%. However, after increasing the Eap from 0.5V to 0.8 the biogas production rate and quality of the CSTR-MEC reactor decreased significantly due to the increase in the pH values i.e pH = 8.2. It was attempted to adjust the pH to neutral pH but the reactor did not recuperate and the pH continued increasing. The quantity and quality of the biogas in the Control reactor kept constant for the four phases. However, the biogas production in the 'CSTR-MEC' reactor dropped almost completely and the methane concentration decreased from 50% to less than 10%. During

the first three phases, the concentration of sulfide in the 'CSTR-MEC' reactor was on average 30% lower than in the 'Control' reactor (Figure 22). During Phase D, the Eap was increased from about 0.5V to about 0.8v, the concentration of sulfide was at the beginning significantly lower in the CSTR-MEC due to the higher Eap applied. Surprisingly, at an Ep of only 0.8V the stainless steel anode started to dissolve from elemental iron to dissolved forms i.e. Fe+2 and Fe+3 and solid forms (Figure 23a and 23b). We are currently analyzing the precipitates on the surface of the cathode as well as the anode products. Precipitates were observed on the surface of the cathode in the 'CSTR-MEC' reactor (Figure 23e). This precipitates covered the whole surface of the electrode and were significantly different than for the Control reactor (Figure 23c vs Figure 23d). The concentration of total ammonium nitrogen (TAN) and the concentration of total phosphorous (TP) in the effluent of the 'CSTR-MEC' reactor were 30% lower than the 'Control' reactor during Phase A and part of Phase B (Figure 24). However, during the rest of Phase B to Phase D, there were no clear differences regarding TAN and TP values which might indicate that the cathode surface is saturated with precipitates and the maximum capacity of precipitating nitrogen and phosphorous salts has been reached. During Phase A to C, the Eap applied was stable averaging 512 ±7.5mV (Figure 25a). However, during phase D, the Eap was difficult to control and behaving unstable averaging 829 ±97mV (Figure 25a). Increasing the HRT does not have a severe impact on the anode potential, cathode potential and power consumption. Yet, the cathode potential decreases by the end of Phase C from -684 ±55mV vs NHE (relative to normal hydrogen electrode) to 830 ±30mV vs NHE (Figure 25c). Both the high instability of the Eap and increase of cathode potential values might indicate saturation of the cathode with precipitates. A clear change in the anode potentials was observed when comparing Phase A-C vs Phase D. The anode potential during Phase A-C was stable averaging +212 ±60mV vs NHE (Figure 25d). During Phase D the anode potential decreased to +85 ±68mV vs NHE. This high variation in the anode potential seems to indicate the dissolving of the anode material. The current density values during Phase A-C were on average 10 ±5mA/L. However, during Phase D, the current density values were the highest i.e. 50 ±27mA/L reactor. Only during Phase A to Phase C this current density represents an energy consumption of about 0.1 ±0.06 kWh/m3 reactor.

The progress of the project continues according to schedule. A lab-scale prototype with a working volume of 20 L was built and it has recently started operation. The design has updated from original proposed design (Figure 21) due to potential clogging. The new design increased the electrodes pair from 3 to 5 (Figure 26). Yet, it was aimed to have the highest electrode area without changing the hydraulics of the system. The reactor was built from transparent poly acrylic using stainless steel (SS 430) as electrode material. The reactor was seeded with anaerobic sludge filling 10% of the reactor volume. Synthetic wastewater is being fed with a hydraulic retention time (HRT) of 4 days under anaerobic mesophilic conditions $(35^{\circ}C \pm 2^{\circ}C)$. The synthetic wastewater has been formulated to simulate the physico-chemical characteristics of a mid-strength domestic wastewater. The lab-scale prototype is currently operated with an Eap of 0.5V. Depending of the results after two months of operation an increase of the Eap will be considered to a 0.8V. Furthermore, another system will be built and will be operated at lower temperature i.e. $15^{\circ}C \pm 2^{\circ}C$. The decrease of temperature will allow us to study the potential application without the need to increase the temperature to accelerate the biological breakdown of organic matter and production of methane. In addition, the performance of these two 20 L will be compared with other technologies available. i.e. High rate activated sludge system. The comparison in terms of process performance and feasibility will be carried out.

Project Status as of January 1, 2017:

The progress of the project is on schedule. We are operating a demo 20-L reactor treating domestic wastewater in our lab in order to gain more knowledge about the hydraulics of this kind of larger reactors. In addition, we are trying to improve the bio-electrochemical parameters in such a system (i.e. applied voltage and electrode potentials). We operated the 20-L reactor for a period of 150 days in four different phases. We have learnt that this reactor takes about 30 days to stabilize or reach a steady state (start-up). During the operation of this reactor, we are focusing on the cathode reactions and on wastewater composition in order to optimize the crystallization of phosphate salts on the surface of the cathode. We tried different potentials (i.e. from 0.5 to 2.8 Volts) and different anode material. Overall, we have seen an increase of bio-degradation of organic matter at higher and lower voltages. However, we have not seen a clear removal of phosphate during the operation of the demo reactor. We are planning to add magnesium, calcium and iron salts in order to enhance phosphate salt precipitation in the reactor. Finally, we will try to slowly release the anode material to achieve precipitation of phosphate due to iron release. From January 2017, we are planning to collect real wastewater and operate in a trailer on site with a newly build 20 liter demo reactor under lower temperatures i.e. $15^{\circ}C \pm 2^{\circ}C$. In addition, the performance of these two 20 L reactors will be compared with other technologies available, i.e. high rate activated sludge system. The comparison in terms of process performance and techno-economical analysis will be carried out.

Project Status as of June 30, 2017:

Two 20-L scale prototypes of microbial electrochemical septic tanks were operated in lab setting for about 120 days at two temperatures, 15 °C and 25 °C. Overall, the results confirmed that the tanks are capable of removing organic matter and phosphorous at both temperatures. The project further decided to move to pilot-scale testing (100-gal tank with an 80-gal liquid holding capacity) and successfully demonstrated the technology application in close-to-real operating conditions of typical septic tanks in Minnesota. To make the large scale demo possible, relevant resources were appropriately allocated through five discussion sessions with septic tank expert and wastewater process engineers. The pilot-scale testing has been running for three stages for 80 days, i.e., the control, applied with 0.82 V and 1.13 V. The two treatment stages achieved phosphorus removal efficiency of 28.2% and 41.6%, respectively. As a comparison, the control condition otherwise released 15% of phosphorus from the inoculum. An economic assessment on the technology installation and operation was conducted based on the parameter inputs from pilot-scale testing. When the average net present cost of having a 1000-gal septic tank for 30-year in Minneapolis area is \$7795, the replacement of the conventional septic tank by the microbial electrochemical septic tank increased the cost by 37%, with an estimated net present cost of \$10661. When the alternative system was powered by solar panel, the net present cost will be \$10345, or 33% increase from the cost of having a conventional system. All the experimental and economic analysis results will be shared with and disseminated to potential users and experts through relevant workshops, the PI's lab website, and possibly the Onsite Sewage Treatment Program of UMN.

Overall Project Outcomes and Results:

Septic systems treat sewage generated from homes and mid-sized facilities that do not have access to centralized wastewater treatment plants. They are effective in retraining and degrading organic matters in the sewage, however, the tanks do not substantially decrease phosphorus nutrients in effluent. The nutrients may enter surrounding aquatic environment and have a negative impact in water bodies such as lakes and rivers. To overcome the limitation, we developed a novel septic system that is better at removing phosphorus and organic matter than conventional septic tanks by using a "microbial electrolytic cell" which uses electricity to promote biological reactions inside the tank. Different reactor designs, sizes and operational parameters were investigated, i.e., from laboratory scale to pilot scale. Laboratory scale experiments were carried out at the University of Minnesota - Department of Bioproducts and Biosystems Engineering and the pilot scale was tested at the Saint Paul Municipal Wastewater Treatment Plant of Metropolitan Council Environmental Services. A preliminary techno-economic assessment showed that around one third additional installation cost is needed to implement this technology, and this enables 50%-90% of the phosphorus removal from the sewage, compared to the conventional septic system. The collaboration with the Metropolitan Council Environmental Services led to the development of a new technology that can help communities effectively manage their wastewater treatment systems and improve public health and the environment. The technology developed from this project, together with the information obtained from the techno-economic analysis, could be useful to thousands of rural communities and help decision-making process of trying the novel tank configuration. When communities effectively manage their wastewater treatment systems, public health and the environment are adequately protected while the community has the management structure in place over the long-term.

IV. PROJECT ACTIVITIES AND OUTCOMES:

ACTIVITY 1: Reactor optimization at lab-scale level

Description: The aim of this activity is to find the most efficient reactor design and operation in terms of quality of the effluent, nutrient recuperation and biogas production. To achieve this, laboratory scale continuously stirred tank reactors will be designed and built with working volumes of approximately 2 liters (Outcome 1). The CSTRs will be used as the septic tank part of a coupled system.

Lab-scale prototype systems will be studied, primarily focusing on evaluating different electrode materials and understanding mechanisms for the increased biogas production and phosphorus crystallization in order to finalize the best reactor design (Outcome 2). Electrochemical analysis will be conducted to screen suitable cathode materials of the smallest onset potential and internal resistance, the largest exchange current density, and the least inhibition induced by the real domestic sewage. Different approaches will be followed based on the coupled system to understand mechanisms for the biogas production. Possible promoting factors for increased biogas production will be screened out, including the increased hydrogen partial pressure induced by MEC cathode, the increased attached biomass, and the improved electro-activity of anaerobic granules. The phenomenon and mechanism for phosphorous (or phosphate) crystallization on cathode surface will be evaluated through designed experiments and appropriate characterization methods.

Optimization for the coupled system will be carried out in order to achieve better quality effluent, nutrient recuperation and biogas production (Outcome 3). Major operational variables include the MEC applied voltage, hydraulic retention time, and medium temperature. These variables will be evaluated by experimental design and analyzed by response surface methodology for their effects on reactor performance of biogas production rate, methane production rate, hydrogen sulfide concentration, COD removal, total phosphorus removal, and total nitrogen removal. Chemical and physicochemical characteristics of the influent and effluents will be analyzed by recommended methods or APHA-AWWA Standard Methods, and compared with statewide standards.

| Summary Budget Information for Activity 1: | ENRTF Budget: | \$ 124,106 | | |
|---|---------------|-------------|--|--|
| | Amount Spent: | \$ 124,106 | | |
| | Balance: | \$ 0 | | |
| Activity Completion Date: Jan1 th , 2016 | | | | |

| Outcome | Completion Date | Budget |
|--|-----------------------------|----------|
| 1. Lab scale septic tank design and construction | Oct 1 st , 2014 | \$24,851 |
| 2. The best reactor design has been chosen | June 1 st , 2015 | \$52,936 |
| 3. The operational parameters have been optimized. | Jan 1 st , 2016 | \$46,319 |

Activity Status as of January 1, 2015:

Several types of reactor vessels were prepared in lab to simulate septic tanks. The reactors include five up-flow column reactors in Figure 1 (~ 150 mL of volume of each reactor), about 20 serum bottle reactors in Figure 2 (150 mL and 1.5 L), 18 air-cathode reactors in Figure 3 (30 mL), and two CSTRs made by using the commercial fermentation bioreactors in Figure 4 (1.6 L). Different research objectives will be evaluated by using different reactor types. The up-flow column reactors allow the insertion of electrodes with different electrode surface areas. The serum bottle reactors are easy to manage air-tight condition so that the biogas volume and quality can be precisely measured and analyzed. The air-cathode will allow the cathode precipitate (phosphate salts) formation without external power supply, and will be tested to compare the precipitation kinetics with anaerobic condition. The CSTR reactors were made from the commercial bioreactors so that they will guarantee a precise control on temperature and agitation speed, which are important parameters in reactor performance. Reference 600 Potentiostat (Gamry Instruments, Warminster, PA) was purchased with Echem Analyst software and Dummy Cell 4 for electrochemical analysis. This equipment will allow experimentation for cyclic voltammetry, electrochemical impedance spectroscopy, chronoamperometry, and some other electrochemical analysis. Based on the prepared reactors and the electrochemical analytic equipment, Activity 2 will be conducted to quantify and optimize the mass change of anode and cathode in septic tanks, electrode activities for redox reactions and nutrient recuperation, and biogas production rate and yield.



Figure 1. Up-flow column reactors. A and B, overview of the reactors and peripheries; and C, D, and E, electrodes after operations.



Figure 2. Serum bottle reactors. A and B, 150 mL reactors with media and electrode couples; and C, 1.5 L reactor vessels.



Figure 3. Air-cathode reactors. A, end-view; and B, top-view.



Figure 4. CSTR reactors prepared from fermenters. A, vessel holder and control systems; and B, CSTR in operation.



Figure 5. Reference 600 potentiostat.

Activity Status as of July 1, 2015:

The research plan stated that the objective of this activity was to choose the best reactor design. Completed works and results are listed as follows.





Figure 6. Cyclic voltammograms in phosphate buffer solution (10 mM, pH=7): A, SS 304 10; B, SS 316 10; C, SS 430 10; D, SS 304 4; and E, carbon cloth.



Figure 7. Cyclic voltammograms in phosphate buffer solution with Na2S (5 mM): A, SS 304 10; B, SS 316 10; C, SS 430 10; D, SS 304 4; and E, carbon cloth.

Objective 1: electrodes selection

The primary goal for electrode selection is to select a material with a strong catalytic effect for hydrogen evolution reaction (HER), which can stimulate the creation of pH gradient and hydrogen production. The best scenario for the anode reaction is that it's being minimized, only to allow microbial oxidation of organic matter so that interruption on microorganisms can be ruled out. The second best scenario is that it should proceed to favor water oxidation rather than the oxidation of electrode material, in order to avoid wasting electrode. Stainless steel has recently been evaluated for its application for both as anode and cathode in microbial electrochemical systems [1]. Given similar or superior anode and cathode catalytic effects to other materials, stainless steel could have a potential to replace carbon-based materials (graphite of different morphologies, e.g., cloth, brush, rod and plate) with its additional advantages: cost-effective, easy to handle, and chemically stable. Cyclic voltammetry was scanned at scanning rates ranging from 10 mV/s to 2000 mV/s for several stainless steel materials (SS 304, 316, and 430) and carbon cloth in several different media (phosphate buffer solution at pH 7, the buffer with Na₂S, and dairy manure). The number following the SS type, e.g., 10, indicates the mesh number of the electrode, and electrodes were cut to have a dimension of 14.5 cm × 4 cm = 58 cm². Potentiostat Reference 600 (Gamry) and Ag/AgCl reference electrode (3 M, +207 vs. standard hydrogen electrode) was used to obtain the CV scan task. The voltammograms are displayed as follows (Figure 6, 7, and 8):



Figure 8. Cyclic voltammograms in manure: A, SS 304 10; B, SS 316 10; C, SS 430 10; D, SS 304 4; and E, carbon cloth.

From cyclic voltammetry, it can be seen from the current of hydrogen adsorption/desorption region at neutral pH that SS 430 displayed the best catalytic effect on HER reaction, and is the most suitable electrode material.

Objective 2: kinetics tests

Reactors were made in laboratory for testing. Based on CV results, stainless steel 430 was used for both anode and cathode. Reactor configuration was changed from fermenter-type to box-type, in order to place electrode with more surface area (Figure 9). For the kinetic test, simulated septic tanks were only fed with sewage and inocula at the beginning, and reactors were applied with voltage between 0.25 V to 1.50 V with increment of 0.25 V. Current densities were recorded three times during operation of 25 days and averaged and shown in Figure 10. Some parameters of the reactors are listed in Table 1.



Figure 9. Illustration (A) and photos of simulated septic tanks (B).

Table 1. Configuration and operating parameters of the electrode assisted septic tanks

| Volume: 0.5 L (headspace: 0.5 L) |
|---|
| Electrode pairs: 6 pairs |
| Anode/cathode surface area: $6 \times 40 = 240 \text{ cm}^2$ |
| Sludge and sewage: 20 mL + 480 mL (sludge: maintained AD; sewage: blue lake wwtp) |
| VSS ratio in the inoculated sewage: anaerobic sludge/sewage: 3:1 |
| Inoculation duration: 25 d |
| Temperature: 16 °C |
| Voltage: stated above (current in the upper-right figure) |
| Liquid sampling: every 5 days |
| Gas sampling: in gas sampler bags (reactors air-tight) |



Figure 10. Current density in each voltage levels (0.25 V to 1.50 V with a 0.25 V increment).

During the initial settling of particulates from inoculated sewage, it can be see that reactive and total phosphorus, total COD, and total nitrogen experienced an initial separation from suspension to bottom sediment (Figure 11). Ammoniacal nitrogen was mostly remained in the liquid phase.



Figure 11. Initial separation of nutrients due to the sedimentation of particulates.

The whole media including solids in reactors were sampled after 25 days' operation (Figure 12). The samples were tested for their total solids, volatile solids, total suspended solid, and volatile suspended solids levels. From reactor #0 to reactor #3 (0 V to 0.75 V), the total solids or volatile solids either had a small reduction or a slight increase. However, starting from reactor #4, both types of solids were substantially increased. The same phenomena were observed for the suspended types of solids as well. The settled sludge volume in 100 mL of sample after 0.5 h was increased significantly in reactor #4 to #6; meanwhile, the settling capability of solids, sludge volume index was obtained, and the reactors #5 and #6 had more dense solids, which was a results of anode release of iron and nickel oxide/hydroxide over the operation under the voltage greater than 1.25 V which triggered the anode pitting/corrosion and coupled the cathode reaction of water reduction, anodic biofilm may not be developed or functions deactivated due to lack of organic nutrients in the later stage of operation.

Liquid samples were also obtained every five days, and analyzed for the evolution of nutrients and COD. In the control reactor, both total and reactive phosphorus had a gradual increase until the day 10. After that, phosphorus level kept relative stable. It was not clear why phosphorus experienced an initial increase, and the possible reasons are that polyphosphate is released from bacterial intracellular components under anaerobic digestion, and that calcium or magnesium bound phosphate is released from inoculum which was obtained from dairy manure fed anaerobic digesters. However, except for reactor #1 and #2, the other four reactors removed phosphorus at a higher rate than its release rate, and on day 10, most P was removed from liquid medium. A higher voltage achieved a higher removal efficiency. For voltage between 0.75 V and 1.0 V, operating septic tank for 5 days is able to reduce phosphorus to be less than 2.2 mg-P/L. Again, with a full development of anodic biofilm, it is expected that phosphorus removal can be increased. The other nutrients properties were given in Figure 14. So the next studies in the continuous mode were operated at voltage levels of 0.50 V, 0.63 V, 0.75 V, and 0.88 V, with the too high voltage ruled out because of too active anodic reactions of electrode corrosion and water oxidation which changed the composition of settled solids and anode biofilm.



Figure 12. Solids properties of influent and effluent of the reactors.



Figure 13. Phosphorus evolution during 25 days' operation of the reactors.



Figure 14. Nutrients profiles during operation and gas generation.

Objective 3: continuous operation

Continuous operation is currently carried out for 13 reactors (0.7 L) with different treatment: two levels of temperature, five levels of voltage, and with chlortetracycline addition (an antimicrobial; 1 mg/L). Reactors were maintained in batch fed mode with a hydraulic retention time (HRT) of 7 days and substrate replacement every 3.5 days (0.35 L). Liquid was sampled every day and biogas was sampled and analyzed every three HRT cycles. The phosphorus levels of the liquid samples are given in Table 2, showing substantial phosphorus removal. However, the exact mechanism for phosphorus removal is not elucidated yet. The reactors will be maintained for another 6 HRTs with conditions changed to favor biogas production (increasing inoculum and tripping headspace air), and will later be subject to cyclic voltammetry analysis and electrode potential analysis to suggest the biofilm growth on anode and evaluate the effect of biofilm on phosphorus removal.

Table 2. Total phosphorus levels for the influent and effluent of electrode-assisted reactors

| | Treatment | | | Total P on the sampling day, mg/L | | | | | | | |
|-----------|-----------|------------|----------------------------|-----------------------------------|------|-------|------|------|------|------|------|
| | | | | Inf | Eff | Inf | Eff | Inf | Eff | Inf | Eff |
| Reactor # | Temp, ⁰C | Voltage, V | CTC contaminant, 1 mg/L | 0 | 6 | 6 | 10 | 10 | 14 | 14 | 17 |
| 1 | 25 | 0 | No | 15.04 | 8.13 | 10.69 | 9.01 | 7.85 | 8.12 | 8.42 | 9.99 |
| 2 | 25 | 0.5 | No | 15.04 | 0.19 | 10.69 | 0.17 | 7.85 | 0.79 | 8.42 | 0.34 |
| 3 | 25 | 0.63 | No | 15.04 | 0.18 | 10.69 | 0.2 | 7.85 | 0.7 | 8.42 | 1.02 |
| 4 | 25 | 0.75 | No | 15.04 | 0.1 | 10.69 | 0.06 | 7.85 | 0.37 | 8.42 | 0.25 |
| 5 | 25 | 0.88 | No | 15.04 | 0.15 | 10.69 | 0.12 | 7.85 | 0.24 | 8.42 | 0.11 |
| 6 | 15 | 0 | No | 15.04 | 8.97 | 10.69 | 9.39 | 7.85 | 7.96 | 8.42 | 8.1 |
| 7 | 15 | 0 | Yes | 15.04 | 8.7 | 10.69 | 9.28 | 7.85 | 7.63 | 8.42 | 8.76 |
| 8 | 15 | 0.5 | No | 15.04 | 0.59 | 10.69 | 0.4 | 7.85 | 0.54 | 8.42 | 1.79 |
| 9 | 15 | 0.63 | No | 15.04 | 0.21 | 10.69 | 0.2 | 7.85 | 0.4 | 8.42 | 1.97 |
| 10 | 15 | 0.75 | No | 15.04 | 0.13 | 10.69 | 0.27 | 7.85 | 0.17 | 8.42 | 1.25 |

| 11 | 15 | 0.88 | No | 15.04 | 0.12 | 10.69 | 0.09 | 7.85 | 0.78 | 8.42 | 0.79 |
|----|----|------|-----|-------|------|-------|------|------|------|------|------|
| 12 | 15 | 0.75 | Yes | 15.04 | 0.05 | 10.69 | 0.17 | 7.85 | 0.12 | 8.42 | 0.67 |
| 13 | 15 | 0.88 | Yes | 15.04 | 0.07 | 10.69 | 0.05 | 7.85 | 0 | 8.42 | 0 |

Activity Status as of January 1, 2016:

Two continue stirred tank reactors (CSTR's) termed 'Control' reactor and 'CSTR-MEC' reactor with a working volume of 1.6 L were prepared (Fig. 15). Both reactors are operated in continuous mode under mesophilic conditions ($35^{\circ}C \pm 2^{\circ}C$) at a hydraulic retention time of 32 ± 0.2 days and at organic loading rates (OLR) of 0.9 ± 0.04 gCOD/L.day.

Figure 15. Experiment set-up of CSTR. Control reactor is operated in open circuit (no power supply)



Both reactors have an anode projected area of 308 cm² and a cathode projected area of 375 cm². The electrode material chosen is stainless steel (SS 430) since it has shown the best catalytic effect in previous work. The Control reactor is operated in open circuit mode and the CSTR-MEC reactor is operated with an applied voltage (Eap) of 0.52 ±0.01 Volts. The reactors were fed continuously with dairy manure as model substrate with constant agitation. The reference electrode allows constant measurement of the electrode potentials during the whole experimental period using a datalogger. In addition, the datalogger allows the recording of current densities and Eap. Biogas production was recorded using the water displacement principle and biogas quality was analyzed using a gas chromatographer. The reactor was inoculated with anaerobic sludge in about 10 gVS/L reactor and operated during a start-up period (about 3 weeks) to ensure that the bacterial community is healthy and the COD consumption and biogas production are appropriate (data not shown).

Table 3. Physico-chemical characteristic of the influent of the CSTR reactors

| рН | 7.85 ±0.28 |
|-----------------------------------|-------------|
| Total COD (g O ₂ /L) | 25.6 ±2.6 |
| Soluble COD (g O ₂ /L) | 6.5 ±0.3 |
| Total VFA (mg CH₃COOH/L) | 1446 ±50 |
| Total Nitrogen (mg N/L) | 974.3 ±44.2 |
| Total Ammonia-Nitrogen (mg N/L) | 279.8 ±8.7 |
| Total Phosphorus (mg P/L) | 233.5 ±9.6 |
| Soluble Phosphorus (mg P/L) | 103.2 ±8.5 |

Table 3 shows the characteristics of the substrate used in this experiment. The substrate contains high concentrations of total chemical oxygen demand (COD) in about 25 gO_2/L . The soluble fraction of COD accounted for about 25% of the Total COD, which is comparable with fresh substrate as shown by the low concentration of short chain fatty acids (VFA) i.e. 1.4 gCH3COOH/L. The COD:N:P ratio of the substrate indicates that the substrate can be subjected to anaerobic digestion. Although there are high fractions of nitrogen and phosphorous, these concentrations will not be inhibitory for the anaerobic microorganism. This model substrate was chosen because of its high concentrations of phosphorous and nitrogen.

Table 4. Reactors performance.

| | Control | CSTR-MEC |
|---|-------------|-------------|
| HRT (days) | 32.0 ±0.2 | 32.2 ±0.2 |
| Eap (V) | - | 0.52 ±0.01 |
| OLR (gCOD/L.d) | 0.9 ±0.04 | 0.9 ±0.04 |
| Biogas (L/L.d) | 0.19 ±0.01 | 0.19 ±0.01 |
| Conversion (%) | 41.5% ±2.6% | 43.0% ±2.6% |
| Methane, CH ₄ (%) | 61.9% ±1.1% | 61.9% ±1.3% |
| Hydrogen sulfide, H ₂ S (ppmv) | 255 ±117 | 113 ±80 |

With an HRT of about 32 days for both reactors and an Eap of about 0.5V for the CSTR-MEC, the total COD recovered as biogas was in the same order of magnitude in both reactors i.e. $41.5 \pm 2.6\%$ for the Control reactor and $43.0\% \pm 2.6\%$ for the CSTR-MEC reactor as shown in Table 4. Moreover, the methane (CH₄) concentration in the biogas was similar in both reactors i.e. about 61%. Yet, the concentration of hydrogen sulfide (H₂S) was about 50% lower in the CSTR-MEC reactor. Interestingly, the amount of total ammonium nitrogen (TAN) and total phosphorous in the effluents of the CSTR-MEC reactor is about 20% lower than the Control reactor indicating that struvite (NH₄MgPO₄) might be precipitating in the reactor. It might be possible that the struvite is formed on the surface of the cathode. This has to be confirmed by opening the reactor at the end of the operational period. The cathode potential recorded at an Eap of about 0.5V reached a maximum average of -750mV vs NHE (relative to normal hydrogen electrode). Additionally, the current density value was at the highest around 18 mA/L reactor. Cathode potential and current density fluctuated during the operational period probably due to the presence of biodegradable COD following feeding patterns. Both cathode potential and current density values were comparable with other studies using other electrode materials such as carbon cloth. Yet, no effects on phosphate and H₂S were reported.

Figure 16. Cathode potential and current density of the CSTR-MEC reactor.



The preliminary work in this study indicates that the CSTR-MEC is capable of removing H₂S from the biogas and removing phosphorous and nitrogen from the effluent (Table 4 and Table 5). For instance, TAN concentration in

the effluent of the CSTR-MEC is about 20% less than in the effluent of the control reactor. This evidence might suggest that struvite might be precipitated in the reactor. However, this has to be confirmed at the end of the operation of the reactors when the mass balances are closed.

| | Control | CSTR-MEC |
|---------------------------------|---------------|--------------|
| рН | 7.33 ±0.02 | 7.39 ±0.03 |
| Conductivity (mS/cm) | 8.5 ±0.9 | 9.6 ±1.2 |
| Alkalinity (mg-CaCO3/L) | 1171.6 ±130.9 | 1171.5 ±97.9 |
| Total solids (g/L) | 11.3 ±2.2 | 9.8 ±1.6 |
| Ash (g/L) | 4.9 ±1.0 | 4.9 ±0.9 |
| Suspended solids (g/L) | 5.6 ±2.3 | 4.5 ±1.4 |
| Total COD (g-O2/L) | 12.3 ±2.9 | 8.5 ±1.8 |
| Soluble COD (g-O2/L) | 1.9 ±0.8 | 1.2 ±0.3 |
| Total VFA (mg-CH3COOH/L) | 231.2 ±106.6 | 223.5 ±81.6 |
| Sulfide (mg-S/L) | 1.7 ±2.0 | 0.6 ±0.6 |
| Total Nitrogen (mg-N/L) | 967.8 ±156.1 | 789 ±61.1 |
| Total Ammonia-Nitrogen (mg-N/L) | 602.3 ±70.8 | 455.4 ±44.0 |
| Total Phosphorus (mg-P/L) | 247.3 ±115.4 | 146.5 ±38.9 |
| Soluble Phosphorus (mg-P/L) | 27.7 ±7.9 | 27.7 ±5.4 |

Table 5. Physico-chemical characteristic of the effluents of the CSTR reactors (Control and CSTR-MEC)

After more than 30 days of operation, the characteristics of the reactors' effluents, i.e. unchanging pH values and non-accumulation of intermediates VFA such as butyric acid, show that the process has reached a steady state and that the anaerobic digestion process is stable (Table 4).

| , , | | • |
|----------------------------|--------------|-------------|
| | Control | CSTR-MEC |
| Acetic (mg CH3COOH/L) | 139.7 ±80.4 | 134.2 ±55.3 |
| Propionic (mg CH3COOH/L) | 80.3 ±42.8 | 67.3 ±32.4 |
| lso-butiric (mg CH3COOH/L) | 0.1 ±0.3 | 0.2 ±0.4 |
| Butyric (mg CH3COOH/L) | 0.8 ±0.9 | 3.2 ±9.0 |
| Iso-valeric (mg CH3COOH/L) | 1.0 ±1.3 | 2.4 ±6.6 |
| Valeric (mg CH3COOH/L) | 0.2 ±0.5 | 0.2 ±2.1 |
| Iso-caproic (mg CH3COOH/L) | 5 ±4.1 | 3.5 ±2.7 |
| Caprioic (mg CH3COOH/L) | 3.3 ±3.0 | 3.5 ±2.1 |
| Heptanoic (mg CH3COOH/L) | 0.7 ±0.4 | 0.6 ±0.3 |
| Total VFA (mg CH3COOH/L) | 231.2 ±106.6 | 223.5 ±81.6 |

Table 4. Short chain fatty acid profile of the effluents of the CSTR reactors (Control and CSTR-MEC)

In the following months, other operational parameters will be studied such as OLR, HRT and Eap. In addition, mass balances will be carried out to estimate the removal efficiencies of the system. At the end of the operation of the reactors, linear sweep voltammetry (LSV) and cyclic voltammetry (CV) will be performed to collect valuable information on the electron transfer mechanisms in the reactors.



Figure 17. Total phosphorus in the influent and effluent of the reactors operated at different applied voltage and temperatures.

Objective 2: sulfide removal performance and other nutrients

Five samples were analyzed for sulfate and sulfide concentrations (Table 4 and Figure 25). Results indicates sulfate-reducing process generated sulfide in the control reactors; however, electrode-assisted reactors contained significantly less amounts of sulfide.

| | 2015/9/11 | | 2015/9/18 | | 2015/9/28 | | 2015/11/1 | | 2015/11/8 | |
|----------------|-----------|---------|-----------|---------|-----------|---------|-----------|---------|-----------|---------|
| | Sulfate | Sulfide |
| | mM | mM |
| Inf | 0.779 | 0.003 | 0.867 | 0.019 | 0.883 | 0.000 | 0.676 | 0.002 | 0.677 | 0.030 |
| Control, 25 oC | 0.134 | 0.250 | 0.134 | 0.061 | 0.086 | 0.074 | 0.337 | 0.313 | 0.166 | 0.168 |
| 0.50 V, 25 oC | 0.117 | 0.000 | 0.117 | 0.000 | 0.091 | 0.000 | 0.118 | 0.000 | 0.028 | 0.000 |
| 0.63 V, 25 oC | 0.038 | 0.000 | 0.038 | 0.000 | 0.066 | 0.000 | 0.044 | 0.000 | 0.027 | 0.000 |
| 0.75 V, 25 oC | 0.073 | 0.000 | 0.073 | 0.000 | 0.109 | 0.000 | 0.058 | 0.000 | 0.067 | 0.000 |
| 0.88 V, 25 oC | 0.051 | 0.000 | 0.051 | 0.000 | 0.076 | 0.000 | 0.033 | 0.001 | 0.039 | 0.000 |
| Control, 15 oC | 0.085 | 0.029 | 0.085 | 0.011 | 0.121 | 0.044 | 0.137 | 0.134 | 0.073 | 0.084 |
| 0.50 V, 15 oC | 0.290 | 0.118 | 0.290 | 0.005 | 0.382 | 0.035 | 0.175 | 0.063 | 0.169 | 0.000 |
| 0.63 V, 15 oC | 0.099 | 0.029 | 0.099 | 0.000 | 0.124 | 0.000 | 0.070 | 0.000 | 0.030 | 0.000 |
| 0.75 V, 15 oC | 0.115 | 0.015 | 0.115 | 0.000 | 0.107 | 0.000 | 0.076 | 0.000 | 0.034 | 0.000 |
| 0.88 V, 15 oC | 0.074 | 0.000 | 0.074 | 0.000 | 0.064 | 0.000 | 0.080 | 0.001 | 0.045 | 0.000 |

Table 6. Results on sulfate and sulfide analysis for five samples

Figure 18. Sulfate and sulfide concentrations in the influent sewage, control reactor, and electrode-assisted reactors.

Major nitrogen components analysis did not show substantial effect in ammoniacal nitrogen concentration (Figure 26). Influent COD was substantially reduced in digestion process; however, voltage application did not display any noticeable difference from the control groups (Figure 27).



Figure 19. Ammonium nitrogen and total nitrogen concentrations in the influent sewage, control reactor, and electrode-assisted reactors averaged from five feeding cycles.



Figure 20. COD concentrations in the influent sewage

Activity Status as of July 1, 2016:

Continuing previous activities, two continue stirred tank reactors (CSTR's) termed 'Control' reactor and 'CSTR-MEC' reactor with a working volume of 1.6 L were operated in continuous mode under mesophilic conditions (35°C ±2°C) for a period of 160 days (Fig. 15) in four operational phases:

| Table 7. Operational phases | | | | | | | |
|-----------------------------|-----------|---------|---------|----------|--|--|--|
| | Phase A | Phase B | Phase C | Phase D | | | |
| HRT (days) | 32.0 ±0.2 | 25 ±0.4 | 20 ±3.0 | 20 ±0.5 | | | |
| Eap (V)* | 0.5 ±0.01 | | | 0.8 ±0.1 | | | |

*Only for the CSTR-MEC reactor. Control reactor was operated in open circuit during the whole experimentation

By changing the operational parameters such as HRT and Eap, the stability of the process was studied. During the first three phases, the HRT was decreased from about 30 to 25 days in Phase B to 20 days in Phase C and D. As expected, the amount of biogas produced by each reactor increased due to the increase of the organic loading rate as depicted by Figure 22. However, clear differences between both reactors in terms of biogas production, biogas composition and conversions efficiency were not observed. The conversion efficiencies during phase A to C were in the same order of magnitude for both reactors i.e. about 40%. However, after increasing the Eap from 0.5V to 0.8 during Phase D, the biogas production rate and quality of the CSTR-MEC reactor decreased significantly due to the increase in the pH values i.e pH = 8.2. It was attempted to adjust the pH to neutral pH but the reactor did not recuperate and the pH continued increasing. The quantity and quality of the biogas in the Control reactor

kept constant for the four phases. However, the biogas production in the 'CSTR-MEC' reactor dropped almost completely and the methane concentration decreased from 50% to less than 10%.



Figure 22. Biogas production rate (liters of biogas per liter of reactor per day) in both reactors, 'Control' reactor and CSTR-MEC reactor (R2). Sulfide concentration in the effluents in both reactors, 'Control' reactor and CSTR-MEC reactor (R2).

High hydrogen sulfide (H₂S) gas concentrations are considered an issue due to potential corrosion of the septic system and health risk. The concentration of aqueous sulfide (HS⁻) was monitored during the whole experiment as an indirect measurement of H₂S gas. Aqueous HS⁻ is in equilibrium with H₂S gas (HS⁻ \leftrightarrow H₂S_{aq} \leftrightarrow H₂S_{gas}). During Phase A to C, the concentration of sulfide in the 'CSTR-MEC' reactor was on average 30% lower than in the 'Control' reactor (Figure 22). During Phase D, the concentration of sulfide was at the beginning significantly lower in the CSTR-MEC due to the higher Eap applied. Surprisingly, at an Ep of only 0.8V the stainless steel anode started to dissolve from elemental iron to dissolved forms i.e. Fe⁺² and Fe⁺³ and solid forms (Figure 23a and 23b). It seems that the electrochemical oxidation of iron in the anode might produce monomeric species (i.e. Fe(OH)₃) and polymeric hydroxy complexes which are also known as hydrous ferric oxides, HFO (i.e. Fe(H₂O)₆₃⁺, Fe(H₂O)₅(OH)₂⁺, Fe(H₂O)₄(OH)₂⁺, Fe₂(H₂O)₈(OH)₂₄⁺ and Fe₂(H₂O)₆(OH)₄₄⁺ which are highly porous and have high surface areas with adsorption capabilities. We are currently analyzing the precipitates on the surface of the cathode as well as the anode products.



Figure 23. Visual observation of the electrodes after operating the reactors for about 160 days. (a) Anode in the CSTR-MEC reactor; (b) Anodic production of iron precipitates in the CSTR-MEC reactor; (c) Cathode in the Control reactor; (d) Cathode in the CSTR-MEC reactor

Precipitates were observed on the surface of the cathode in the 'CSTR-MEC' reactor (Figure 23e). The precipitates covered the whole surface of the electrode and were significantly different than for the Control reactor (Figure 23c vs Figure 23d). The concentration of total ammonium nitrogen (TAN) and the concentration of total phosphorous (TP) in the effluent of the 'CSTR-MEC' reactor were 30% lower than the 'Control' reactor during Phase A and part of Phase B (Figure 24). However, during the rest of Phase B to Phase D, there were no clear differences regarding TAN and TP values which might indicate that the cathode surface is saturated with precipitates and the maximum capacity of precipitating nitrogen and phosphorous salts has been reached.



Figure 24. (a) Total ammonium nitrogen (TAN) concentration in the effluent of both reactors 'Control' reactor and CSTR-MEC reactor (R2). (b) Total phosphorous in the effluent of both reactors 'Control' reactor and CSTR-MEC reactor (R2).

The installation of a reference electrode with a datalogger allowed constant measurement of the electrode potentials and current densities during the whole experimental period. During Phase A to C, the Eap applied was stable averaging 512 ±7.5mV (Figure 25a). However, during phase D, the Eap was difficult to control and behaving unstable averaging 829 ±97mV (Figure 25a). It can be observed from Figure 25 that increasing the HRT does not have a severe impact on the anode potential, cathode potential and power consumption. Yet, the cathode potential decreases by the end of Phase C from -684 ±55mV vs NHE (relative to normal hydrogen electrode) to 830 ±30mV vs NHE (Figure 25c). Both the high instability of the Eap and increase of cathode potential values might indicate saturation of the cathode with precipitates. A clear change in the anode potentials was observed when comparing Phase A-C vs Phase D. The anode potential during Phase A-C was stable averaging +212 ±60mV vs NHE (Figure 25d). During Phase D the anode potential decreased to +85 ±68mV vs NHE. This high variation in the anode potential seems to indicate the dissolving of the anode material.

The current density values during Phase A-C were on average 10 \pm 5mA/L. However, during Phase D, the current density values were the highest i.e. 50 \pm 27mA/L reactor. Only during Phase A to Phase C this current density represents an energy consumption of about 0.1 \pm 0.06 kWh/m³ reactor. This power consumption can be calculated to be about 15% of the total potential energy recovery (based on embodied energy from methane production).



Figure 25. Visual observation of the electrodes after operating the reactors for about 160 days. (a) Anode in the CSTR-MEC reactor; (b) Anodic production of iron precipitates in the CSTR-MEC reactor; (c) Cathode in the Control reactor; (d) Cathode in the CSTR-MEC reactor

Activity Status as of January 1, 2017:

Continuing the previous lab study. The precipitates observed on the surface of the cathode in the 'CSTR-MEC' reactor (Figure 23e) were analyzed with a scanning electrode microscopy coupled with energy dispersive X-ray analysis (SEM-EDS). SEM pictures confirmed the formation of crystals on the surface of the anode (Figure 26a). The picture shows that the crystals are small and amorphous. Quantitative EDS analysis indicates that the main components of the crystals are oxygen, calcium, iron and phosphorous. The higher concentration of calcium suggests that the presence of calcium (Ca⁺²) and carbonates (CO₃⁻²) ions interact with other elements such as phosphate. From the chemistry composition, it is unlikely that struvite is a major compound due to the presence of large amounts of Ca. It could be possible that other forms such as Ca oxalate or hydroxylapatite are also present following; $5Ca^{+2}+3PO_4^{-3}+H_2O \rightarrow Ca_5(PO_4)_3OH+H^+$ and $Ca^{+2}+CO_3^{-2}\rightarrow CaCO_3$. To properly identify the compounds, XRD analysis is currently carried out. XRD patterns will be compared with database models to properly identify the compounds present in the crystals.



Figure 26. (a) SEM image of crystals on cathode surface; (b) Elemental surface analysis by EDS.

One of the main mechanisms of phosphorus removal is the iron release from the anode due to electrochemical dissolution of the sacrificial anode (stainless steel). Phosphate precipitation with iron (Fe) is commonly used as phosphorous removal technology in water treatment and it follows $Fe^{+2} + PO_4^{-3} \rightarrow FePO_4$. In addition, other compounds, common in stainless steel, such as Chromium (Cr) will be released as well. Interestingly, the crystals formed in the cathode surface contain Fe and Cr in about 9.6% and 0.2% respectively (Table 8). There is a potential to use this technology to clean waste streams polluted with heavy metals.

| Table 8. | Elemental | composition | by EDS | analysis |
|----------|-----------|-------------|--------|----------|
| | | | | |

| | Percentage | | | |
|---------|------------|--|--|--|
| Element | (%) | | | |
| С | 5.8 ±2.6 | | | |
| 0 | 40.7 ±12.8 | | | |
| Na | 0.2 ±0.04 | | | |
| Mg | 2.3 ±1.1 | | | |
| Al | 0.3 ±0.1 | | | |
| Si | 3.1 ±3.9 | | | |
| Р | 6.0 ±3.4 | | | |
| S | 0.6 ±0.4 | | | |
| Cl | 0.3 ±0.1 | | | |
| К | 0.3 ±0.3 | | | |
| Са | 30.5 ±10.3 | | | |
| | 24 | | | |

| Cr | 0.2 ±0.1 |
|----|-----------|
| Mn | 0.6 ±0.3 |
| Fe | 9.6 ±10.2 |

We are currently preparing a manuscript including these experiments. We will include the XRD analysis in the manuscript and next report.

Activity Status as of June 30, 2017:

As reported in previous months another 20-liter lab-scale demo reactor was build in order to compare the performance of our system at two operational temperatures. One reactor was operated at $15^{\circ}C \pm 2^{\circ}C$ and another reactor was operated at $25^{\circ}C \pm 2^{\circ}C$ for a period of about 120 days. Both reactors were fed with real wastewater from the St. Paul wastewater treatment plant operated by the Metropolitan Council Environmental Services. Both reactors were constructed as previous described (Figure 26) and operated with an average HRT of 4.0 days with different patterns of applied voltage, Eap (Table 12 and Table 13).

| Fable 12. Operational parameters of the 20-lif | er lab-scale prototype rea | ctor operated at 15°C ±2°C |
|--|----------------------------|----------------------------|
|--|----------------------------|----------------------------|

| | Phase I | Phase II | Phase III | Phase IV | Phase V | Phase VI | Phase VII | Phase VIII | Phase IX |
|---|---------|----------|-----------|----------|---------|----------|-----------|------------|----------|
| Days | 5 | 15 | 10 | 10 | 10 | 10 | 10 | 20 | 15 |
| Applied voltage (E _{ap} =V) | - | 0.88 | 1.0 | 1.1 | 1.3 | 1.4 | 1.5 | 1.6 | 1.7 |

The stepwise increase of Eap was aimed to increase the removal efficiency of phosphorous (P). It can be observed that Eap for lower temperatures (i.e. 15°C) is higher to achieved the same level of P removal.

| Table 13. Operational parameters of the zo-internab-scale prototype reactor operated at $z_3 \in \pm z$ c | | | | | | | | |
|--|---------|----------|-----------|----------|---------|----------|-----------|--|
| | Phase I | Phase II | Phase III | Phase IV | Phase V | Phase VI | Phase VII | |
| Days | 5 | 15 | 10 | 10 | 10 | 15 | 10 | |
| Applied voltage (E _{ap} =V) | - | 0.88 | 1.0 | 1.1 | 1.3 | 1.1 | 1.0 | |

Table 13. Operational parameters of the 20-liter lab-scale prototype reactor operated at 25°C ±2°C

Overall the results show that both systems are capable of removing organic matter and phosphorous (Figure 30). The reactor operated at 15°C reached the highest chemical oxygen demand (COD) removal (about of 52% of total COD and about 37% of soluble COD) with an Eap of about 1.7 Volts. At this Eap, the highest phosphorous removal was also observed (from day 106 onwards, Phase IX). The total P removal was about 61% and the soluble P removal was about 65%. The total solids removal reached was about 20% (Figure 30a). At these conditions, the effluent quality of the reactor operated at 15°C was on average 195 mg/L of total COD, 104 mg/L of soluble COD, 2.7 mg/L of total P, 1.7 mg/L of soluble P and 810 mg/L of total solids. The reactor operated at 25°C reached the highest COD removal (about of 68% of total COD and about 56% of soluble COD) with an Eap of between 1.3 and 1.1 Volts. At this Eap the highest phosphorous removal was also observed (from day 60 to day 100, Phase V and Phase VI). The total P removal was about 54% and the soluble P removal was about 86%. The total solids removal reached was about 12% (Figure 31a). At these conditions, the effluent quality of the reactor operated at 25°C was on average 102 mg/L of total COD, 61 mg/L of soluble COD, 2.5 mg/L of total P, 0.5 mg/L of soluble P and 1000 mg/L of total solids. After phase VI the Eap was decreased further in order to fine tune the Eap required for the highest P removal for the system. The removal efficiencies decreased when the Eap was set to around 1 V. At an Eap of 1V, the removal efficiencies were about 62% of total COD, 66% of soluble COD, 35% of total P and 50% of soluble Ρ.



Figure 30. Evolution of the influent and effluent concentrations in the 20-liter lab-scale prototype reactors operated at 15 °C and 25 °C; (a) total chemical oxygen demand; total COD (b) soluble chemical oxygen demand; Soluble COD; (c) total phosphorous, total P and (d) soluble phosphorous, soluble P.

The total ammonium nitrogen (TAN) concentration in the effluent of the reactor operated at 15 °C was about 40%



(a)

(d)

26

Figure 31. Evolution of the influent and effluent concentrations in the 20-liter lab-scale prototype reactors operated at 15 °C and 25 °C; (a) total solids and (b) total ammonium nitrogen, TAN.

The daily energy consumption of the reactors was about 0.2 KWh/m³_{reactor} for the reactor operated at Eap of about 1.7 Volts (15 °C) and between 0.09-0.04 KWh/m³_{reactor} for the reactor operated at Eap of between 1.3 and 1.1 Volts (25 °C). The reactor operated a lower temperature had a higher electricity requirements. The electricity requirement when operating the reactor at 15 °C was about double than operating at 25 °C. The main objective of the lab-scale experiments is to find the most efficient reactor design and operational parameters in terms of quality the effluent. It was found that the efficiency of the process is temperature dependent. When operating the reactor at 15 °C, the most appropriate Eap seems to be 1.7 V to achieve the best quality of the effluent (the lower phosphorous and the lower organic content). When operating reactors at 25 °C, the most suitable Eap seems to be between 1.1-1.3 V. These values were taking into account when operating the pilot demo reactor.

Final Report Summary:

The Activity 1 aimed to design, operate, and optimize lab-scale microbial electrochemical septic tank for targeted objectives of on-site wastewater treatment improvement. This activity used one-liter and 20-liter tanks and tested various stainless steel materials for better treatment performance, and eventually came up with stainless steel AISI 430 and 304. The one-liter scale tanks proved the phosphorus removal (between 20.7% and 98.3% of removal efficiency at 0.5 to 0.88 V treatments) and recovery from sewage via the improved phosphorus precipitation on electrodes and the increased phosphorus amount settled in sludge. A relatively lower voltage, e.g., 0.5 and 0.63 V retained a higher proportion of phosphorus on electrodes than the proportions achieved with a higher voltage application, e.g., 0.75 and 0.88 V. The further increasing the applied voltage may generate more sludge volume in septic tank. Methane production in microbial electrochemical septic tank was boosted by microbial electrochemical treatment, and the largest difference was found to be occurred at 0.88 V, increased from 180 mL by 107% at 25 °C, and from 15 mL by 360% at 15 °C. If the produced methane is properly collected and utilized as a potential bioenergy source, the substantially increased methane production in microbial electrochemical septic tank was the advantageous. A preliminary prototype of 20-liter tanks were thereafter built and tested. It was confirmed that, by optimizing voltage range, electrode materials and surface areas, microbial electrochemical septic tank is a promising alternative to the conventional septic tank.

ACTIVITY 2: Prototype construction and evaluation

Description: The main objective is to design and construct an MEC prototype that can be installed on a typical traditional septic tank. A MNDOT community septic tank system will be chosen to test the prototype. The manhole of the septic tank system will be retrofit to accommodate the MEC and the prototype will be primarily built on this existing system to test different operation conditions on the biogas production and nutrient recuperation (Outcome 4). Design considerations and operational parameters will be based on the knowledge acquired from activity 1. Influent flow rates, organic loading rates and hydraulic retention times will be similar to the values of current systems. Chemical and physicochemical characteristics of the influent and effluents as well as crystal properties will be determined according to recommended methods or APHA-AWWA Standard Methods. The operation of the reactors will be carried out in long operational periods to simulate real systems (4-6 months) and parameters such as electric voltage will be adjusted to reach the optimized operation (Outcome 5).

| Summary Budget Information for Activity 2: | ENRTF Budget: | \$ 92,191 |
|--|---------------|-------------|
| | Amount Spent: | \$ 92,191 |
| | Balance: | \$ 0 |
| Activity Completion Date: June 30th, 2017 | | |

| Outcome | Completion Date | Budget | | |
|----------------------------|-----------------------------|-----------|--|--|
| 4. Prototype construction. | June 1 st , 2016 | \$ 53,595 | | |

| 5. Prototype optimization. | Jan 1 st , 2017 | \$ 38,596 |
|----------------------------|----------------------------|-----------|

Activity Status as of January 1, 2015:

We start to purchase some chemicals and supplies to prepare for the activity 2.

Activity Status as of July 1, 2015:

We start to purchase some chemicals and supplies to prepare for the activity 2.

Activity Status as of January 1, 2016:

A lab-scale prototype with a working volume of 20 L is being built. The reactor will have a high projected electrode area per unit of reactor (Figure 21). The aim of this reactor is to provide the highest electrode area in order to facilitate the precipitation of phosphorous salts. The electrode material will be stainless steel (SS 430) as this was proven to be the most appropriate in previous experiments. The prototype is being built in the St. Paul Campus Machine workshop.

Figure 21. Lab-scale prototype set-up.



Activity Status as of July 1, 2016:

A lab-scale prototype with a working volume of 20 L was built and it has recently started operated. The design has changed from original proposed design (Figure 21) due to potential clogging. The new design increased the electrodes pair from 3 to 5 (Figure 26). Yet, it was aimed to have the highest electrode area without changing the hydraulics of the system. The reactor was built from transparent poly acrylic using stainless steel (SS 430) as electrode material. Biogas production is being recorded using water displacement principal a biogas composition is being analyzed using a MicroGC. The reactor was seeded with anaerobic sludge filling 10% of the reactor volume. Synthetic wastewater is being fed with a hydraulic retention time (HRT) of 4 days under anaerobic mesophilic conditions (35°C ±2°C). Septic systems are designed to operate with HRT of about 2 days. However, the reactor volume is usually over dimensioned. Thus, practical HRT values are closer to 4 days. The synthetic wastewater has been formulated to simulate the physico-chemical characteristics of a mid-strength domestic waste water. The wastewater is prepared using primary sludge collected from Blue Lake wastewater treatment plant (Shakopee, Minnesota) as a source of suspended solids. Other soluble components were provided as reported in literature. The lab-scale prototype is currently operated with an applied voltage (Eap) of 0.5V. Depending of the results after two months of operation an increase of the Eap will be considered to a 0.8V. Furthermore, another system will be built and will be operated at lower temperature i.e. 15°C ±2°C. The decrease of temperature will allow us to study the potential application without the need to increase the temperature to accelerate the biological breakdown of organic matter and production of methane. In addition, the performance of these two 20 L will be compared with other technologies available. i.e. High rate activated sludge system. The comparison in terms of process performance and feasibility will be carried out.

Figure 26. Lab-scale prototype set-up drawing and photos.



Activity Status as of January 1, 2017:

We continue the activities with the 20-liter demo reactor in our lab (Figure 26). We are currently operating one 20-liter reactor with an HRT of 4.0 \pm 0.1 days under mesophilic temperature 15°C \pm 2°C in 4 phases (Table 9). The four phases included; operating the reactor without power applied (Eap) for about 60 days (Phase I), operating with an Eap of 0.5V (phase II) and 0.6V (phase III) for about 30 days in each Phase. During Phase IV, we operated the reactor at Eap of about 2.8V using only 280 cm² of cathode-projected area and as anode we used a titanium anode rode. The objective of this phase was to evaluate the capacity of the cathode to precipitate salts on their surface alone, ultimately avoiding sacrificing the anode material. This high voltage was necessary to achieve high cathode potentials >-700 mV vs NHE.

Table 9. Operational parameters of the 20-liter prototype reactor. Phase I includes the start-up of the reactor.

| | Phase I | Phase II | Phase III | Phase IV |
|---|----------|----------|-----------|----------|
| Temperature (°C) | 30 ±2 | | | |
| HRT (days) | 4.0 ±0.1 | | | |
| Projected electrode area (cm ²) | 900 | | | 180 |
| Days | 60 | 30 | 30 | 20 |
| Applied voltage (Eap=V) | - | 0.5 | 0.6 | 2.8 |

We fed the reactor with synthetic wastewater simulating mid-strength domestic wastewater as shown in Table 10. During the four phases, the pH on the reactor kept neutral (Table 10). Conductivity values indicate that mineralization of salts due to biological activity occurs in all phases. The increase of conductivity in the effluent during phase I and phase III was about 10%. During phase II and phase IV, the effluent conductivity increased in about 15% higher than phase I and III. The removal of total solids in the demo reactor during phase I was on average about 10%. During phase IV, the solids removal was on average about 25%. The total and soluble COD removal in all phases was in the same order of magnitude averaging around 70% and 50% (Table 10 and Figure 27a and b).

Table 10. Overall performance of the 20-liter prototype reactor

| Influent | Phase I | Phase II | Phase III | Phase IV | |
|----------|---------|----------|-----------|----------|--|
|----------|---------|----------|-----------|----------|--|

| рН | 8.3 ±0.3 | 7.4 ±0.2 | 7.2 ±0.3 | 7.4 ±0.1 | 7.1 ±0.1 |
|-----------------------------------|----------|----------|-----------|----------|----------|
| Conductivity (uS/cm) | 915 ±75 | 1041 ±18 | 1320 ±263 | 1076 ±72 | 1334 ±96 |
| Total solids (mg/L) | 776 ±136 | 711 ±192 | 509 ±53 | 553 ±102 | 691 ±240 |
| Total COD (mgO ₂ /L) | 701 ±130 | 232 ±57 | 297 ±35 | 260 ±39 | 223 ±34 |
| Soluble COD (mgO ₂ /L) | 347 ±115 | 118 ±52 | 194 ±24 | 175 ±12 | 191 ±68 |
| Total Ammonium Nitrogen (mg/L) | 32 ±8 | 41 ±4 | 54 ±6 | 45 ±3 | 41 ±5 |
| Total Kjendahl Nitrogen (mg/L) | 67 ±7 | 58 ±3 | 64 ±8 | 61 ±3 | 68 ±1 |
| Total Phosphorous (mg/L) | 13 ±3 | 13 ±2 | 14 ±3 | 13 ±3 | 12 ±2 |
| Soluble Phosphorous (mg/L) | 8 ±2 | 12 ±1 | 14 ±1 | 11 ±1 | 10 ±1 |

The total ammonium nitrogen (TAN) concentration in the effluent of the reactor was about 20% higher during Phase I. TAN values were about 50% and 40% higher during Phase II and Phase III. During Phase IV, the increase of TAN in the effluent was one order of magnitude. The much higher value of TAN during Phase IV indicates that the biodegradation is higher when using titanium electrodes at higher Eap (Figure 27c). However, the effluent concentrations of total phosphorous were in the same order of magnitude removal during the four phases as shown by Figure 27d. Thus the precipitation of struvite salts on the cathode surface was not occurring. Struvite usually precipitates as stable white orthorhombic crystals in a 1:1:1 molar ratio of Mg^{+2} :NH₄⁺⁴:PO₄⁻³. In order to verify that Mg^{+2} was present in the influent, the influent and effluent were analyzed using ICP (Table 11).



Figure 27. Evolution of the influent and effluent concentrations in the 20-liter prototype reactor; (a) total chemical oxygen demand; CODtotal; (b) total ammonium nitrogen, TAN; (c) total phosphorous, P_{total} and (d) total soluble phosphorous, P_{soluble}

The elemental analysis of the influents and effluents using inductive coupled plasma (ICP) showed that the concentration of Mg^{+2} in the influent and effluent was in the same order of magnitude confirming that struvite was not precipitated under the conditions studied (Table 11). Yet, the concentration of Mg^{+2} (i.e. 2 mg/L) might be lower to fully precipitate all the phosphorous in the wastewater. Thus, relying solely on the precipitation of struvite like salts on the cathode would not be enough to achieve high phosphorous removal efficiencies. We are currently investigating the possibility of adding more Mg^{+2} or Fe⁺² salts to optimize the removal of phosphorous.

Table 11. Elemental analysis of influent and effluent of the 20-liter prototype reactor

| Al | 1.0 ±0.2 | 0.4 ±0.1 |
|------|------------------|------------------|
| В | 0.2 ±0.01 | 0.1 ±0.02 |
| Ca | 17.9 ±0.7 | 15.5 ±0.5 |
| Cd | BDL [*] | BDL* |
| Cr | BDL [*] | BDL [*] |
| Cu | 0.1 ±0.01 | 0.05 ±0.01 |
| Fe | 1.3 ±0.3 | 0.5 ±0.1 |
| К | 9.9 ±1.0 | 10.6 ±0.9 |
| Mg | 2.2 ±0.6 | 1.8 ±0.3 |
| Mn | 0.1 ±0.01 | 0.05 ±0.01 |
| Na | 31.2 ±1.3 | 31.4 ±1.4 |
| Ni | BDL | BDL |
| Р | 5.0 ±1.3 | 5.0 ±0.9 |
| Pb | BDL | BDL |
| Zn | 0.2 ±0.03 | 0.1 ±0.02 |
| **** | 1 | |

BDL, Below detection limit

Figure 28 shows the electrochemical values during the four phases. The Eap started after about 64 days (Figure 28a). Prior to that time, the reactor was operated in open circuit without Eap. During Phase II, the Eap was 0.50 ±0.02 V, the current density average was about 1.5 mA/L_{reactor} (Figure 28b), the cathode potential average was -670 mV vs NHE (Figure 28c), and the anode potential was about +64 mV vs NHE (Figure 28d). At the beginning of Phase II, the system had a perturbation due to the short circuit of one of the cells creating high perturbation in the current density and potentials. After the short circuit was fixed, the Eap was 0.66 ±0.1 V, the current density was on average 0.46 mA/L_{reactor} (Figure 28b), the cathode potential average was -304 mV vs NHE (Figure 28c), and the anode potential was about +269 mV vs NHE (Figure 28d). The increase on Eap from 0.5 to 0.6 had a effect on both the cathode and the anode potentials with more impact in the anode. Interestingly, a decrease was observed on the current density in about three orders of magnitude regardless of the increase of Eap. We wanted to evaluate the capacity of the cathode alone to precipitate phosphorous salts. Therefore, during Phase IV, we introduced titanium rode as anode material in order to increase the cathode potential. At an Eap of about 2.8 V, the observed cathode potentials reached a maximum of about -650mV vs NHE (Figure 28c). The anode potential values reached were about +2V, which is the maximum recommended for this material. During this phase we did not see an increase of the removal efficiency of phosphorous. Thus, for this type of reactor and wastewater, increasing the Eap would not necessarily increase the removal of phosphorous from the cathode alone. The maximum potential required to sacrifice the anode is about +0.447 V vs NHE following Fe -> Fe⁺² + 2e⁻. This value could be reached with an Eap above 0.7V.







Figure 28. (a) Applied voltage versus time, Eap; (b) Current density per liter reactor versus time; (c) cathode potentials versus time and (d) anode potentials versus time.

We are currently operating the reactor with small additions of calcium, magnesium and iron salts in order to promote the precipitation of phosphate salts (i.e. struvite, hydroxyapatite, etc). Furthermore, we have constructed another demo reactor. The new reactor will be operated at a lower temperature, i.e. 15°C ±2°C using real wastewater. We are planning to collect real wastewater from any of the following three locations (i) the University of Minnesota housing sewage collection system; (ii) a resting facility close to the metro area operated by MnDOT (Minnesota Department of Transportation), or (iii) from a wastewater treatment plant in the St. Paul area operated by the Metropolitan Council. Ideally, we will locate a trailer close to the water source that will serve as an onsite laboratory. The trailer will be equipped with the basic laboratory hardware functioning with an independent generator for energy and heat requirements. The sewage will be collected from the water collection point into the equalization tank using a centrifugal pump (Figure 29). Influent samples will be taken from the equalization tank will feed the demo septic tank at a specific hydraulic retention time using a peristaltic pump. The effluents of the demo tank will be collected in the effluent tank for sampling. Once the effluent tank is full, the treated wastewater will be pumped back to the water collection point.



Figure 29. Process chart for the demonstration experiments.

In the eventuality of not being able to park our trailer close to the water collection point, fresh wastewater will be collected using a one cubic meter IBC and brought to our department laboratory on a weekly basis. We will place the demonstration laboratory in our building in a special space where demonstration experiments are carried out. In addition to the performance of the demo reactors, other technologies available, such as high rate activated sludge system, will be operated and compared with our proposed system. The comparison in terms of process performance and feasibility will be carried out.

Activity Status as of June 30, 2017:

The project further decided to move to pilot-scale testing stage and successfully demonstrated the technology application in close-to-real operating conditions of typical septic tanks in Minnesota. During the pilot-scale testing, 100-gal HDPE tank with a working volume of 80-gal was operated for 78 days treating raw wastewater (Figure 32a and 32b). The tank was modified to resemble a septic system (i.e. influent and effluent baffles were built). The electrode material was stainless steel AISI 430 with a total electrode surface area of 7.8 m², a design based on results of bench scale experiment. The designated voltage treatment scheme over the experiment was 0 V (control), 0.88 V and 1.1 V (Figure 33). Sewage pumps with microcontrollers connected with relays were installed to provide an average hydraulic retention time of approximately 3.3 days (Figure 34). The tank was housed in a sampler with the temperature controlled at the targeted value of around 20 °C at St Paul wastewater treatment

plant operated by the Metropolitan Council Environmental Services (MCES). The tank was inoculated with anaerobic sludge to a volume of about 10% of working volume of the tank, and fed with sewage. Anaerobic sludge was collected from Blue Lake wastewater treatment plant (Shakopee, Minnesota), and sewage was collected onsite from the waterway of primary wastewater. Water samples, i.e., the influent and effluent of the tank, were collected and analyzed on each work day for water characteristics including pH, conductivity, sulfide, total solids, total suspended solids, total ammonium nitrogen, total phosphorus, reactive phosphorus, total soluble phosphorus, and total particulate phosphorus, total COD and soluble COD.



Figure 32. The process diagram (A), photo of septic tank (B), pump controlling system (C), and voltage application and monitoring system (D).

After 30-day operation under control conditions, the effluent phosphorus and total suspended solids content kept relatively stable in the latest four days' samples. Voltage was thereafter applied to electrodes of the septic tank at 0.84 V. However, based on the following 8 days results, the monitored electrical current was not as large as expected and appeared too low (Table 14; Figure 33), and phosphorus profiles were not improved after treatment. It was suspected that the corrosions might have happened to electrode surface that was subject to sewage corrosion and inhibited anodic/cathodic reactions. A temporary treatment with higher voltage (1.5 to 2 V) was applied to electrodes to strip off electrodes deposits, and then the voltage was recovered to the designated value starting day 41. After the treatment, the electrical current reading was improved and phosphorus removal started to be observed.

| Stages | | Applied voltage, V | Electrical current, A | Power consumption, W | |
|--------|----------------------------|-----------------------|--------------------------|-------------------------|-------------|
| | | | Mean ± SD | Mean ± SD | Mean ± SD |
| 0 | Control | Day 0-30 | 0 | 0 | 0 |
| 1 | V= 0.84 | Day 29-38 | 0.84 ± 0.14 | 0.29 ± 0.9 | 0.28 ± 1.48 |
| 2 | V=0.82 (electrode treated) | Day 41-58 | 0.82 ± 0.27 | 0.81 ± 0.45 | 0.78 ± 0.78 |
| 3 | V= 1.13 | Day 65-75 | 1.13 ± 0.07 | 1.65 ± 0.17 | 1.86 ± 0.15 |

Table 14. Scheme of voltage application for the electrode-assisted pilot-scale septic tank



Figure 33. The applied voltage (A), monitored electrical current (B), and power consumption (C) in the electrode-assisted septic tank.



Figure 34. The hydraulic retention time (A) and experimental site room temperature (B).

Time course profiles of the results at different treatment stages were illustrated in Figures 35-38. Data summaries for each stage of stable operation were listed in Tables 15-18. The effluent pH and conductivity were close to the influent, and were within the range of without creating environmental hazards (Figure 35). Comparisons of phosphorus profiles among the three stages, i.e., the control, 0.82 V, and 1.13 V treatment, suggested that: 1) while in the control condition, total phosphorus was slightly higher (14.9%) in effluent than in influent, the treatment achieved phosphorus removal efficiency of 28.2% and 41.6%; 2) while in the control condition, total soluble phosphorus removal and 56.3% of particulate phosphorus removal (Figure 36). Total solids were removed by 4.9%, 18.1%, and 15.3%, respectively, while total suspended solids, one of the critical parameters influencing septic system performance, were removed by 55.3%, 32.9%, and 54.7% for the three stages, respectively (Figure 37). Both the influent and effluent experienced sudden quality fluctuation in terms of solids, and the fluctuation added additional noise so that it was hard to evaluate if the treatment improved solids removal.



Figure 35. Evolution of the influent and effluent pH (A) and conductivity (B).



Figure 36. Evolution of the influent and effluent phosphorus profiles: total phosphorus (A), total soluble phosphorus (B), total particulate phosphorus (C), and reactive phosphorus (D).



Figure 37. Evolution of the influent and effluent solids profiles: total solids (A), total suspended solids (B), and total dissolved solids (C).

The treatment decreased sulfide content from 1.85 mg/L to 0.85 mg/L, and from 8.32 mg/L to 1.78 mg/L, at 0.82 V and 1.13 V, respectively (Figure 38). However, the average effluent sulfide in the control condition was also lower than the corresponding influent, 0.32 mg/L vs. 1.79 mg/L, respectively. Total ammonium nitrogen experienced an increase of 22.2%, 37.7%, and 21.3% during the three stages, respectively.



Figure 38. Evolution of the influent and effluent total COD (A), soluble COD (B), total sulfide (C), total ammonium nitrogen (D) content.

| Table 15. Wastewater characteristics between da | v 15 and 29 under control condition |
|---|-------------------------------------|
|---|-------------------------------------|

| Day 15 – 29, control | | | | |
|----------------------|-------------|-------------|-----------------------|--|
| | Influent | Effluent | Removal efficiency, % | |
| рН | 7.36 ± 0.3 | 7.31 ± 0.18 | | |
| Conductivity, µS/cm | 1232 ± 144 | 1241 ± 159 | | |
| Sulfide, mg/L | 1.79 ± 2.95 | 0.32 ± 0.29 | 82.0 | |
| TSS, mg/L | 124 ± 36 | 55 ± 21 | 55.3 | |
| TP, mg/L | 5.79 ± 1.74 | 6.65 ± 0.47 | -14.9 | |
| NH4-N, mg/L | 20 ± 4 | 24 ± 7 | -22.2 | |
| RP, mg/L | 3.57 ± 0.87 | 5.78 ± 0.75 | -61.9 | |
| tCOD, mg/L | 398 ± 112 | 199 ± 36 | 50.2 | |
| sCOD, mg/L | 156 ± 38 | 115 ± 31 | 26.4 | |
| TS, mg/L | 1095 ± 189 | 1040 ± 247 | 4.9 | |
| TDS, mg/L | 981 ± 191 | 985 ± 243 | -0.4 | |
| tSP, mg/L | 3.16 ± 0.81 | 4.78 ± 0.91 | -51.3 | |
| tPP, mg/L | 2.63 ± 1.72 | 2.27 ± 1.71 | 13.8 | |

Table 16. Wastewater characteristics between day 30 and 38 under ~0.84 V treatment

| | Day 30 - 38 | | |
|---------------------|-------------|-------------|-----------------------|
| | Influent | Effluent | Removal efficiency, % |
| рН | 7.24 ± 0.2 | 7.17 ± 0.08 | |
| Conductivity, µS/cm | 1548 ± 19 | 1602 ± 58 | |
| Sulfide, mg/L | 1.11 ± 1.56 | 0.33 ± 0.33 | 70.7 |
| TSS, mg/L | 113 ± 30 | 48 ± 15 | 57.6 |
| TP, mg/L | 5.87 ± 0.5 | 6.07 ± 0.05 | -3.4 |
| NH4-N, mg/L | 30 ± 8 | 36 ± 13 | -18.6 |
| RP, mg/L | 4.06 ± 0.54 | 6.32 ± 2.62 | -55.4 |
| tCOD, mg/L | 336 ± 32 | 204 ± 22 | 39.3 |

| sCOD, mg/L | 148 ± 17 | 121 ± 29 | 17.9 |
|------------|-------------|-------------|-------|
| TS, mg/L | 1046 ± 111 | 973 ± 144 | 7.0 |
| TDS, mg/L | 987 ± 64 | 932 ± 134 | 5.5 |
| tSP, mg/L | 3.55 ± 0.67 | 4.26 ± 0.35 | -20.0 |
| tPP, mg/L | 2.25 ± 0.42 | 1.68 ± 0.22 | 25.4 |

Table 17. Wastewater characteristics between day 41 and 58 under ~0.82 V treatment

| | Day 41- 56 | | |
|---------------------|-------------|-------------|-----------------------|
| | Influent | Effluent | Removal efficiency, % |
| рН | 7.34 ± 0.25 | 7.26 ± 0.13 | |
| Conductivity, µS/cm | 1425 ± 70 | 1470 ± 55 | |
| Sulfide, mg/L | 1.85 ± 1.48 | 0.85 ± 1.44 | 54.0 |
| TSS, mg/L | 109 ± 53 | 73 ± 62 | 32.9 |
| TP, mg/L | 6.74 ± 1.85 | 4.84 ± 1.79 | 28.2 |
| NH4-N, mg/L | 27 ± 6 | 37 ± 11 | -37.7 |
| RP, mg/L | 5.81 ± 2.37 | 4.01 ± 0.83 | 30.9 |
| tCOD, mg/L | 436 ± 149 | 163 ± 28 | 62.6 |
| sCOD, mg/L | 168 ± 23 | 89 ± 29 | 47.3 |
| TS, mg/L | 1065 ± 161 | 872 ± 59 | 18.1 |
| TDS, mg/L | 961 ± 143 | 805 ± 87 | 16.3 |
| tSP, mg/L | 4.11 ± 1.15 | 2.78 ± 1.23 | 32.2 |
| tPP, mg/L | 2.63 ± 1.38 | 2.05 ± 0.71 | 22.0 |

Table 18. Wastewater characteristics between day 65 and 75 under ~1.13 V treatment

| | Day 65 - 75 | | |
|---------------------|-------------|-----------------|-----------------------|
| | Influent | Effluent | Removal efficiency, % |
| рН | 7.26 ± 0.12 | 7.07 ± 0.12 | |
| Conductivity, µS/cm | 1522 ± 42 | 1523 ± 28 | |
| Sulfide, mg/L | 8.32 ± 5.99 | 1.78 ± 2.05 | 78.6 |
| TSS, mg/L | 106 ± 45 | 48 ± 18 | 54.7 |
| TP, mg/L | 7.35 ± 1.02 | 4.29 ± 0.51 | 41.6 |
| NH4-N, mg/L | 36 ± 4 | 43 ± 2 | -21.3 |
| RP, mg/L | 6.27 ± 1.46 | 3.8 ± 0.46 | 39.3 |
| tCOD, mg/L | 422 ± 85 | 195 ± 17 | 53.7 |
| sCOD, mg/L | 179 ± 24.35 | 74 ± 25 | 58.4 |
| TS, mg/L | 957 ± 79 | 810 ± 59 | 15.3 |
| TDS, mg/L | 887 ± 28 | 780 ± 48 | 12.1 |
| tSP, mg/L | 4.89 ± 1.14 | 3.21 ± 0.15 | 34.3 |
| tPP, mg/L | 2.47 ± 0.75 | 1.08 ± 0.53 | 56.3 |

Final Report Summary:

The objective of Activity 2 was to construct and evaluate a prototype of microbial electrochemical septic tank for its performance in terms of sewage treatment and other benefits. The prototype reactors were fabricated and tested in lab condition, and the project was further enlarged to pilot-scale testing stage and successfully demonstrated the technology application in close-to-real operating conditions of typical septic tanks in Minnesota. Relevant resources were appropriately allocated through five discussion sessions with septic tank expert and wastewater process engineers. Major progresses included workforce allocation (mechanic, plumber, and electrician), site selection and use contract with Metropolitan Council Environmental Services (MCES), device preparation and installation, sampling and analysis schedule, experimentation and results analysis, a plan for techno-economic assessment, and a plan for dissemination and final reporting. The pilot-scale testing has been running for three stages, i.e., the control, 0.82 V, and 1.13 V. The results suggested that: 1) while in the control condition, total phosphorus was slightly higher in effluent than in influent, the two treatment conditions achieved phosphorus removal efficiency of 28.2% and 41.6%; 2) while in the control condition, total soluble phosphorus

was increased and only a small portion of particulate phosphorus was removed (13.8%), the treatment achieved 34.3% of soluble phosphorus removal and 56.3% of particulate phosphorus removal. The power consumption for the treatment system was 0.78 and 1.86 W for the two treatment conditions. Those results are necessary for conducting a techno-economic assessment and are decisively important when considering the outreach portion of the study – potential users or septic tank experts will be more satisfied and willing to use or introduce this technology as it is proved working for phosphorus removal at the larger scale.

ACTIVITY 3: Economic analysis

An economic assessment will be carried out by considering the cost of the system, the cost related with the operation of the system and the valorization of the outputs such as biogas and nutrients (Outcome 6). Nutrients, recuperated in the form of struvite, have potential as a slow release fertilizer. A comprehensive evaluation of the inputs and outputs of the whole process will be carried out in terms of mass balances, energy balances, and the suitability of outputs as fertilizers. A detailed technological and economic analysis of the proposed system using input data from literature and from the prototype operation will be carried out. The economic analysis will address issues involved in commercial implementation of the system, including the size of facility required for commercial application; the realistic estimates of biogas production rates achievable under commercial conditions; the expected costs to construct a commercial-scale facility; the opportunity cost of capital required; the useful life of the system; the operations and maintenance costs, including labor requirements, repairs, and downtime; and the utilization of the biogas and the value derived from it. These considerations will be incorporated into a discounted cash-flow capital-budgeting analysis that generates results for investment criteria such as net present value, internal rate of return, return on investment, and cash flow surpluses and deficits over time.

| Outrania | Constant of the De | |
|--|--------------------|-------------|
| Activity Completion Date: June 30th, 2017 | | |
| | Balance: | \$ 0 |
| | Amount Spent: | \$ 41,703 |
| Summary Budget Information for Activity 3: | ENRTF Budget: | \$ 41,703 |

| Outcome | Completion Date | Budget |
|--|------------------------------|-----------|
| 6. Evaluation of capital and operational costs | June 30 th , 2017 | \$ 41,703 |

Activity Status as of January 1, 2015:

The activity 3 has not been started yet.

Activity Status as of July 1, 2015:

The activity 3 has not been started yet.

Activity Status as of January 1, 2016:

The activity 3 has not been started yet.

Activity Status as of July 1, 2016:

The activity 3 has not been started yet

Activity Status as of January 1, 2017:

The activity 3 has not been started yet

Activity Status as of June 30, 2017:

This section analyzed and summarized techno-economic results of installing and operating a microbial electrochemical septic tank based on the input from pilot-scale testing with 41.6% of total phosphorus removal. When retrofitting a conventional septic tank to a microbial electrochemical septic tank, the additional cost are generated from the cost of electrode materials, the preparation and installation of electrodes in tanks, energy consumption for voltage application, and potentially increased sludge pump-out frequency. The average cost for

conventional septic tank installation in Minneapolis is estimated to be \$6750 (estimate based on homeadvisor), with the tank itself costs about \$1000 before delivery (e.g., 1000 gallon plastic septic tanks manufactured by Norwesco or Ace Roto-Mold companies). A plastic septic tank can last for between 30 and 40 years before it is necessary to be replaced, and a service life of 30 years is assumed in this analysis. For every three years, septic tank should be pumped for sludge and cleaned. On average, this periodical maintenance costs about \$200 each time in Minneapolis (estimate based on homeadvisor.com). Those costs will be used as baseline values for cost comparison between conventional system and the novel systems of either powered by grid or solar panel. For estimating the additional cost of microbial electrochemical septic tank, the material price is estimated based on worldfreightrates.com) of assuming the material is shipped from Shanghai, CN to Los Angeles, CA, and further delivered to Minneapolis, MN. This approach of material purchase will get a 3-6 folds lower electrode material cost than those being purchased from several other domestic vendors.

Assuming the 1000-gal septic tank is operated at a hydraulic retention time of 3 days and the influent has average total phosphorus content of 6.5 mg/L (the average value over 78 days), the total annual phosphorus input to the tank will be 2.99 kg. Assuming the technology recovers 50% of the phosphorus in solids/sludge phase, the annual production will be 1.49 kg-P, which is equivalent to rock phosphate (~14% P in mineral) of 10.7 kg per year, or \$1.07 value per year assuming a mineral price of \$100/ton at current market. Similarly, when a 100% phosphorus recovery is assumed, the annual phosphorus fertilizer value will be \$2.14. Compared with the effort to recover phosphorus from solids/sludge, and the potential heavy metal concerns, inclusion of the phosphorus value in the analysis is complicated. Nevertheless, this low value of phosphorus does not imply that its environmental concerns can be neglected. The current economic analysis therefore did not monetize the value of the captured phosphorus, and it was well rationalized from the above calculation. We were not able to measure significant biogas production in both the lab-scale experiments and the pilot 100-gal septic system. The valorization of biogas was not included into the techno-economic analysis.

The analysis results are presented in Table 17. For a typical scale of a conventional septic tank, the net present cost of having the treatment facility for 30 years is \$7795, most of which is the capital cost that accounts for 87% of the net present cost. For a microbial electrochemical septic tank that is powered by grid electricity, the total net present cost is \$10661, with the capital cost accounting for 87% as well. If the tank is powered by a 100-Watt solar panel, the cost is slightly decreased to \$10345, with the capital cost accounting for 90%. Therefore, the addition of electrode for better treatment increased the net present cost by 37% and 33%, respectively, depending on the source of power input. This amount of cost increases by 37% and 33% can be acceptable if the better environmental footprint is monetized.

| | Itoms | Conventional | Microbial electrochemical septic tank | | | |
|-----------------|---|--------------|---------------------------------------|------------------|--|--|
| | items | septic tank | Powered by grid | Powered by solar | | |
| | Scale of the targeted septic tank, gal | 1000 | 1000 | 1000 | | |
| Capital cost | Service life time of tanks | 30 | 30 | 30 | | |
| | Cost of installing a conventional septic tank, \$ | 6750 | 6750 | 6750 | | |
| | Required anode and cathode surface area, m ² /tank | - | 100 | 100 | | |
| | Unit price of electrode materials, Alibaba quote, $\ensuremath{\$/m^2}$ | - | 5 | 5 | | |
| | Total cost of electrode materials cost, \$/tank | - | 484 | 484 | | |
| | Material freight charge (Shanghai to Minneapolis), \$/tank | - | 966 | 966 | | |
| | Time for electrode preparation and installation, hr | - | 40 | 40 | | |

Table 17. Techno-economic calculation for a typical 1000-gal microbial electrochemical septic tank installation and operation

| | Labor price \$/br/person | - | 24 | 24 |
|-------|---|------|-------|-------|
| | Electrode preparation and instalation cost, \$/tank | - | 960 | 960 |
| | 100 Watt solar panel price and installation cost | - | - | 60 |
| | Other electronic peripheries and materials cost, \$ | - | 80 | 80 |
| | Total capital cost, \$ | 6750 | 9240 | 9300 |
| | Sludge pump-out and tank maintenance, /year | 0.34 | 0.34 | 0.34 |
| | Cost of tank maintenance for a single time | 200 | 200 | 200 |
| 0&M | Power consumption for powering, W/tank | - | 23 | 23 |
| cost | Energy cost for powering a tank at 12 cents/kWh, \$/year | - | 24 | - |
| | Total annual O&M cost, \$/year | 68 | 92 | 68 |
| Tabal | Interest rate per year, % | 5 | 5 | 5 |
| rotar | Uniform series present cost, \$ | 1045 | 1421 | 1045 |
| lost | Net present cost, \$ | 7795 | 10661 | 10345 |

Final Report Summary:

An economic assessment on the technology installation and operation was conducted based on the inputs from previous two Activities. When the average net present cost of having a 1000-gal septic tank for 30-year in Minneapolis area is \$7795, the replacement of the conventional septic tank by the microbial electrochemical septic tank increased the cost by 37%, with an estimated net present cost of \$10661. When the alternative system was powered by solar panel, the net present cost will be \$10345, or 33% increase from the baseline. The techno-economic analysis results will be shared with and disseminated to potential users and experts in workshops or Onsite Sewage Treatment Program of UMN. The technology developed during this project, together with the information obtained from the techno-economic analysis, could be useful to thousands of rural communities and help decision-making process of trying the novel tank configuration, especially those that do not have access to centralized wastewater treatment facilities. When communities effectively manage their wastewater treatment systems, public health and the environment are adequately protected while the community has the management structure in place to sustainably treat their wastewater over the long-term.

V. DISSEMINATION:

Description: Part of the reactor design in the lab scale, if proved to be innovative, will be applied to the University Office for Technology Commercialization for filling the patent protection. We will publish two to three peerreviewed manuscripts in the related journals to disseminate our results to the general public. We will also use the Onsite Sewage Treatment Program website http://septic.umn.edu/ as one-dissemination mechanism to our targeted audience. The program seeks to protect public health and the environment by improving wastewater treatment through research-based education and outreach for homeowners, small communities, professionals and policy-makers. The technology developed during this project will be posted on the website and it could be useful to thousands of rural communities, especially those that do not have access to centralized wastewater treatment facilities. The primary target to disseminate our research results will be the community based septic tank systems installed in the rural area where multiple family and business are connected to generate relatively large amount of the wastewater. When communities effectively manage their wastewater treatment systems, public health and the environment are adequately protected while the community has the management structure in place to sustainably treat their wastewater over the long-term. The small scale septic tank systems can also be re-designed based on our results to better manage the waste, so the producers of these small scale septic tank systems will also the audience of the technology. Any royalty, copyright, patent, and sales of products and assets resulting from this project will be subject to revenue sharing requirements with ENRTF according to Minnesota Statutes, section 116P.10.

Activity Status as of January 1, 2015:

Some preliminary results have been disseminated through the following conference presentations, and no trust fund has been spent for the travel related to these presentations:

 Hongjian Lin, Carlos Zamalloa, Nicholas Williams, Amelia King, and Bo Hu. Electrochemical Approach for Removal of H2S and Inorganic Nutrients in Anaerobic Digestion. 2014, Poster session of the Department of Bioproducts and Biosystems Engineering, October 29th,St Paul Student Center, University of Minnesota.
 Hongjian Lin, Nicholas Williams, and Bo Hu. Electrochemical Approach for Removal of H2S and Inorganic Nutrients in Anaerobic Digestion. 2014 AIChE Annual Meeting, November 16-21, Atlanta, Georgia.

Activity Status as of July 1, 2015:

No activities for dissemination at this period.

Activity Status as of January 1, 2016:

We made an oral presentation at 2015 ASABE Annual International Meeting:

1. Lin, Hongjian; Zhang, Xin; Gan, Jing; He, Qiyang; Reis, Cristiano R.; Rajendran, Aravindan; Yan, Mi; Yang, Yan; Zhang, Yanmei ; Hu, Bo, Anaerobic digestion with microbial electrochemical systems drives the biogas production, antibiotic removal and nutrient recuperation. Jul 26-29, 2015, New Orleans, Louisiana.

Activity Status as of July 1, 2016:

On July 2016 an oral presentation will be given for the 2016 ASABE Annual International Meeting in Orlando, Florida. The presentation title is: Anaerobic digestion of dairy manure in a continue stirred tank reactor (CSTR) with a microbial electrolysis cell.

Activity Status as of January 1, 2017:

These are the dissemination activities for this period:

- 1. Zamalloa, C., Heger, S. and Hu, B. Novel approaches for rural wastewater treatment: reinventing septic systems (2016) The University of Minnesota Annual BBE Showcase. St. Paul, USA, October 27th, **Poster**.
- 2. Zamalloa, C., Lin, H., Zhang, Xin; Gan, Jing and Hu, B. Anaerobic digestion of dairy manure in a continue stirred tank reactor with a microbial electrolysis cell (2016) Transactions of the ASABE, Orlando, USA. July 17-20, **Presentation.**
- 3. Lin, H., Zamalloa C., and Hu. B Total Phosphorus Removal from Sewage in Microbial Electrochemically Assisted Septic Tanks (2016) AlChE Annual Meeting, San Francisco, USA. November 13-18. **Presentation.**

Activity Status as of June 30, 2017:

One paper was published in a peer-reviewed journal during this half-year cycle:

Lin, H., Liu, W., Zhang, X., Williams, N., & Hu, B. (2017). Microbial electrochemical septic tanks (MESTs): An alternative configuration with improved performance and minimal modifications on conventional septic systems. *Biochemical Engineering Journal*, 120, 146-156.

Final Report Summary:

Project information, results, and major achievements have been disseminated through multiple ways during the project period. **One paper** was published in a peer-reviewed journal:

Lin, H., Liu, W., Zhang, X., Williams, N., & Hu, B. (2017). Microbial electrochemical septic tanks (MESTs): An alternative configuration with improved performance and minimal modifications on conventional septic systems. *Biochemical Engineering Journal*, 120, 146-156.

Four oral presentations were made in 2014 and 2016 AIChE conferences, and in 2015 and 2016 ASABE AIM conferences:

- Lin, H., Nicholas Williams, and Bo Hu. Electrochemical Approach for Removal of H2S and Inorganic Nutrients in Anaerobic Digestion. 2014 AIChE Annual Meeting, November 16-21, Atlanta, Georgia.
- Lin, H.; Zhang, Xin; Gan, Jing; He, Qiyang; Reis, Cristiano R.; Rajendran, Aravindan; Yan, Mi; Yang, Yan; Zhang, Yanmei ; Hu, Bo, Anaerobic digestion with microbial electrochemical systems drives the biogas production, antibiotic removal and nutrient recuperation. ASABE AIM, Jul 26-29, 2015, New Orleans, Louisiana.

- Zamalloa, C., Lin, H., Zhang, Xin; Gan, Jing and Hu, B. Anaerobic digestion of dairy manure in a continue stirred tank reactor with a microbial electrolysis cell (2016) ASABE AIM, Orlando, USA. July 17-20.
- Lin, H., Zamalloa C., and Hu. B Total Phosphorus Removal from Sewage in Microbial Electrochemically Assisted Septic Tanks (2016) AIChE Annual Meeting, San Francisco, USA. November 13-18.

Two posters were presented in BBE Department Showcase:

- Lin, H., Carlos Zamalloa, Nicholas Williams, Amelia King, and Bo Hu. Electrochemical Approach for Removal of H2S and Inorganic Nutrients in Anaerobic Digestion. 2014, Poster session of the Department of Bioproducts and Biosystems Engineering, October 29th, St Paul Student Center, University of Minnesota.
- Zamalloa, C., Heger, S. and Hu, B. Novel approaches for rural wastewater treatment: reinventing septic systems (2016) The University of Minnesota Annual BBE Showcase. St. Paul, USA, October 27th.

Two more manuscripts based on this study are currently under preparation for peer-reviewed journal submission. Besides the academic dissemination, a video of showcasing the pilot-scale testing system was posted in our website (https://bohu.cfans.umn.edu) and youtube (https://z.umn.edu/lccmr-video) for general public access. Important and practical results from this project will also be disseminated in relevant workshops or in Onsite Sewage Treatment Program of UMN.

| A. ENRTF Budget Overview: | | |
|------------------------------------|----------------------------------|---|
| Budget Category | \$ Amount Revised 1/1/2016 | Explanation |
| Personnel: | \$ 212,800 | 1 project manager at 8% FTE for 3 years; 1 extension specialist at 16.7% FTE for 3 years; 1 postdoc researcher at 85% FTE for 3 years. |
| Equipment/Tools/Supplies: | \$22,455 | Chemical, supplies, analysis and lab septic tank systems |
| Capital Expenditures over \$5,000: | \$18,000 | MEC plug-in prototype for the pilot test, part of HPLC purchase |
| Printing: | \$1,035 | Publication cost for the manuscripts |
| Travel Expenses in MN: | \$3,710 | Mileage, lodging, meals for travels to the community septic tank test site for taking samples as well as testing the prototype. U of M plan for travel expense will be used to process the travel cost |
| TOTAL ENRTF BUDGET: | \$258,000 | |

VI. PROJECT BUDGET SUMMARY:

Explanation of Use of Classified Staff: N/A

Explanation of Capital Expenditures Greater Than \$5,000:

\$10,000 is requested to build the MEC plug-in prototype to be set up on the existing septic tank system to test its long term operation. The prototype will be built on the manhole of the septic tank system, and the cover of the manhole will be retrofit to accommodate the pilot sized of MEC, including two electrodes, electric wiring and a hand hole for sampling. This capital equipment is custom made equipment. It will continue to be used throughout its useful life in my research group to test the effectiveness of the electrochemical system to improve the nutrient removal in the Septic tank or other anaerobic digestion systems.

\$8,000 will be spent to contribute a HPLC purchase. It is an Agilent HPLC system equipped with Multiwavelength detector, which will be able to measure some of the components in the wastewater. The HPLC cost is \$31,496.88. LCCMR project is requested to contribute \$8,000 and the rest of the funds for this HPLC purchase will come from Bo Hu's research group. This capital equipment will serve as our primary equipment to analyze water samples; and it will continue to be maintained and used throughout its useful life in the research group as well as the BBE department to analyze samples for environmental remediation types of research, even after the LCCMR project ends in 2017.

Number of Full-time Equivalents (FTE) Directly Funded with this ENRTF Appropriation: 3

Number of Full-time Equivalents (FTE) Estimated to Be Funded through Contracts with this ENRTF Appropriation: 0

B. Other Funds:

| | \$ Amount | \$ Amount | |
|--------------------|-----------|-----------|--|
| Source of Funds | Proposed | Spent | Use of Other Funds |
| Non-state | | | |
| | \$134,160 | \$0 | In-kind services during project period: Unrecovered F&A at 52% MTDC |
| TOTAL OTHER FUNDS: | \$ | \$ | |

VII. PROJECT STRATEGY:

A. Project Partners:

B. Project Impact and Long-term Strategy:

The outcome of the project is to build a supplementary MEC unit that can be plugged into current septic tank systems in order to improve the water quality of septic tanks effluents, recuperate phosphorus nutrient that can be harvested as fertilizer, increase the production and collection of biogas for the bioenergy application and decrease the emissions of GHGs. The project will test the technical feasibility of this new system. In addition, capital and operational costs of the implementation of such a system will be projected and the benefits will be assessed. The research specifically focuses on the modification of large scale septic systems, where collecting the nutrients and biogas is economically possible. It will also provide valuable information to re-design the household septic system with better wastewater treatment. The technology developed during this project could be useful to thousands of rural communities, especially those that do not have access to centralized wastewater treatment facilities. Since half of the MN septic tank systems are actually located in the metro areas, these urban population can also benefit from better wastewater treatment that protects ground and surface waters along with protecting human health. This new information will be reported to existing and new wastewater professionals throughout Minnesota and beyond by our educational program; and it will open the opportunities to promote the applications of the next generation septic system.

C. Spending History:

| Funding Source | M.L. 2008 | M.L. 2009 | M.L. 2010 | M.L. 2011 | M.L. 2013 |
|--------------------------|-----------|-----------|-----------|-----------|-----------|
| | or | or | or | or | or |
| | FY09 | FY10 | FY11 | FY12-13 | FY14 |
| No funds have been spent | 0 | 0 | 0 | 0 | 0 |

VIII. ACQUISITION/RESTORATION LIST:

N/A

IX. VISUAL ELEMENT or MAP(S):

X. ACQUISITION/RESTORATION REQUIREMENTS WORKSHEET: N/A

XI. RESEARCH ADDENDUM:

A separate research addendum has been completed and submitted.

XII. REPORTING REQUIREMENTS:

Periodic work plan status update reports will be submitted no later than January 1, 2015; July 1, 2015; January 1, 2016; July 1, 2016; and January 1, 2017. A final report and associated products will be submitted between June 30 and August 15, 2017.

References:

1. Pocaznoi, D., et al., *Stainless steel is a promising electrode material for anodes of microbial fuel cells.* Energy & Environmental Science, 2012. 5(11): p. 9645-9652.

| Environment and Natural Resources Trust Fund | | | | | | | | | | | | |
|--|---|--------------|-----------------------|----------------------|------------------|-----------------------|----------------------|---|--------------------|---------------------------|------------------|------------------|
| M.L. 2014 Project Budget | | | | | | | | 6 | | | | |
| Project Title: Next Generation Large-Scale Septic Tank System | stems | | | | | | | E | NVIRONMEN | T | | |
| Legal Citation: M.L. 2014, Chp. 226, Sec. 2, Subd. 08g | | | | | | | | A | ND NATURAL RESOURC | ES | | |
| Project Manager: Bo Hu | | | | | | | | | RUSTFOR | | | |
| Organization: University of Minnesota | | | | | | | | | | | | |
| M.L. 2014 ENRTF Appropriation: \$258,000 | | | | | | | | | | | | |
| Project Length and Completion Date: 3 Years, June 30, 207 | 17 | | | | | | | | | | | |
| Date of Report: 7/1/2017 | | | | | | | | | | | | |
| ENVIRONMENT AND NATURAL RESOURCES TRUST FUND BUDGET | Revised Activity 1 Budget 01/01/2017 | Amount Spent | Activity 1 Balance | Activity 2 Budget | Amount Spent | Activity 2 Balance | Activity 3 Budget | Revised Activity 3 Budget 06/30/2017 | Amount Spent | Activity 3 Balance | TOTAL BUDGET | TOTAL BALANCE |
| BUDGET ITEM | | | | Prototype cons | struction and ev | valuation | | Economic and | alysis | | | |
| Personnel (Wages and Benefits) | <u>\$99,205</u> | \$99,205 | \$0 | \$75,359 | \$75,359 | \$0 | \$38,237 | \$35,616 | \$35,616 | \$0 | <u>\$210,180</u> | \$0 |
| Bo Hu, Project Manager: \$34,534 (74.85% salary, 25.15% benefits); 8% FTE for 3 years | | | | | | | | | | | | |
| Sara Heger, Septic system extension specialist: \$45,376 (74.85% salary, 25.15% benefits); 16.7% FTE for 3 years | | | | | | | | | | | | |
| PostDoc Researcher: \$142,890 (83.28% salary, 16.72% benefits); 85% FTE for 3 years | | | | | | | | | | | | |
| Equipment/Tools/Supplies | | | | | | | | | | | | |
| Two lab scale CSTR Septic tank systems (\$2,500 each) | | | | | | | | | | | | |
| Lab supplies: chemicals, tools, bottles, gloves, and lab analysis services | <u>\$14,575</u> | \$14,575 | \$0 | \$5,227 | \$5,227 | \$0 | \$2,653 | \$8,991 | \$8,991 | \$0 | <u>\$28,793</u> | \$0 |
| Capital Expenditures Over \$5,000 | | | | | | | | | | | | |
| A MEC plug-in prototype will be constructed and installed on a typical traditional septic tank, for instance, the MNDOT community septic tank system. The cover of the manhole of the septic tank system will be retrofit to accommodate the pilot sized MEC, including two electrodes, electric wiring and | | | | \$10,000 | \$6,433 | \$3,567 | | \$270 | \$3,837 | -\$3,567 | <u>\$10,270</u> | \$0 |
| a hand hole for sampling. | | | | | | | | | | | | |
| \$8,000 will be spent to contribute a HPLC purchase. It is an Agilent HPLC system equipped with Multiwavelength detector, which will be able to measure some of the components in the wastewater. The HPLC cost is \$31,496.88. LCCMR project is requested to contribute \$8,000 and the rest of the funds for this HPLC purchase will come from Bo Hu's research group. | \$8,000 | \$8,000 | \$0 | | | | | | | | <u>\$8,000</u> | \$0 |
| Printing | | | | | | | | | | | | |
| Publication cost for manuscripts | \$508 | \$10 | \$498 | \$350 | \$0 | \$350 | \$177 | -\$848 | \$0 | -\$848 | <u>\$10</u> | \$0 |
| Travel expenses in Minnesota | * • • • • | <u> </u> | * • • • • • | <u> </u> | | * • • • • | * ~~~ | <u> </u> | A- · · · | * ••• ••• • | A- : - | |
| Mileage, lodging, meals for travels to the community septic tank test site for taking samples as well as testing the prototype. U of M plan for travel expense will be used to process the travel cost | \$1,818 | \$130 | \$1,688 | \$1,255 | \$74 | \$1,181 | \$636 | -\$2,326 | \$544 | -\$2,870 | <u>\$748</u> | \$0 |
| Others | | | | | | | | | | | | |

| COLUMN TOTAL | \$124,106 | \$121,920 | \$2,186 | \$92,191 | \$87,093 | \$5,098 | \$41,703 | \$41,703 | \$48,988 | -\$7,285 | \$258,000 | \$0 |
|--------------|-----------|-----------|---------|----------|----------|---------|----------|----------|----------|----------|-----------|-----|