Ohio University

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Task #5: Removal and Reuse of Phosphorus as Fertilizer from CAFO Runoff

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Table of Contents

Executive Summary:

A process is described for the removal of dissolved orthophosphate and nitrogen from Confined Animal Feeding Operations (CAFO) wastewater in the form of a fertilizer. Currently, CAFO facilities store wastewater in large lagoons, holding upwards of a year's worth of waste. Solids settle to the bottom of the lagoon and are processed off-site while surface water is released. This water contains an average of 20 ppm (mg phosphorus/L) of dissolved orthophosphate and some dissolved nitrogen, in the form of ammonia or ammonium, both of which contribute to eutrophication. By targeting these compounds for removal prior to wastewater release, nutrient pollution can be reduced. Dissolved orthophosphate and ammonium can be removed from wastewater in the form of struvite, a solid phosphorus-based fertilizer.

Struvite, or magnesium ammonium phosphate, can be precipitated from an alkaline solution with the addition of magnesium. Upstream concentration, by reverse osmosis, of the initial 20 ppm solution to around 250 ppm increases struvite yield. Through the use of a continuous electrochemical plug flow reactor (EPFR), a basic solution is created via sacrificial magnesium metal electrodes, which leads to struvite formation. This specific reactor design achieves greater than 90% conversion of orthophosphate to solid struvite, as shown through bench-scale testing, which is filtered from the effluent and used as a slow release phosphorus based fertilizer. Primary benefits of this process include the reduction in eutrophication caused by runoff of nitrogen and phosphorus and the creation of a useful fertilizer. Secondary benefits include reducing the amount of mined phosphate rock that is required to make common fertilizers and is a finite resource, increased wastewater processing rates which lower land usage for storage, and the creation of clean water via reverse osmosis.

North Carolina had been targeted for initial implementation of this technology due to the large number of CAFO facilities it contains. While there are currently limited regulations regarding phosphorus release, the state of North Carolina has a long term goal to implement such regulations which this technology will help facilities meet. The full-scale process is projected to have a 20 m^2 footprint with the capabilities of processing 53.2 m^3/day with low safety concern and minimal required maintenance. The process will cost $21,000 to install/operate, and will make $310 per year based solely on struvite recovery.

Background and Problem Scope:

Excess nitrogen and phosphorus in waterways leading to nutrient pollution is one of the most challenging environmental issues in the modern day.[1] Proper management of nitrogen and phosphorus compounds and their entry into waterways should be carefully monitored to improve and preserve the quality of surface and groundwater reserves. While these elements naturally occur in aquatic ecosystems, an overabundance leads to eutrophication, causing the formation of large algal blooms. Algal blooms produce toxins that pollute drinking water, block out sunlight, and upon decomposition, deplete dissolved oxygen. This is directly taxing on aquatic life, and indirectly on humans who are exposed to those waterways or through consumption of the water or affected aquatic life.[1,2] Nationally, research to combat the hazards associated with eutrophication is becoming more prevalent, specifically with regard to Concentrated Animal Feed Operation (CAFO) facilities.[3]

The National Conference of State Legislatures defines CAFOs as agricultural facilities that house and feed a large number of animals in a confined area.[4] CAFOs produce a large volume of animal waste that is rich in nitrogen and phosphorus compounds and are, therefore, major contributors to nutrient pollution in waterways.[5] While nutrient pollution is a national problem, North Carolina has shown outward concern for eutrophication as the state holds a high number of CAFO facilities.[1] Because of this, implementation of technologies to properly manage phosphorus and nitrogen release from CAFO wastewater streams in North Carolina must become a priority. As of 2018, 88% of the total permitted CAFOs in North Carolina were swine facilities.[6] Thus, targeting swine facilities for nutrient control may act as a sufficient starting point for wide-scale nutrient pollution reduction.[7]

When CAFOs produce large amounts of animal waste, the phosphorus and nitrogen compounds eventually reach major waterways by runoff or by seeping into groundwater.[2] If the dissolved phosphorus could be removed from CAFO waste, the runoff could be drained to waterways with a significant reduction in the risk of nutrient pollution. Furthermore, recovery of phosphorus and nitrogen compounds from CAFO waste as a fertilizer has been investigated. The purpose of a fertilizer is to replace the phosphorus, nitrogen, and trace compounds such as magnesium in soil that is taken up by growing plants.[9] Recovery of phosphorus as a fertilizer is

important because natural phosphorus rock, used for the creation of phosphorus based fertilizers, is expected to be depleted in the next 90 years.[8,9]

Nitrogen present in animal manure is a result of excess nitrogen in animal feed.[10] Animals excrete nitrogen as ammonia, or other non-ammonia forms which are degraded into ammonia by microbes or enzymes present in the manure.[10] Manure also contains significant quantities of inorganic phosphorus which takes the form of orthophosphate (PO_4^{3-}).[11] Ammonium and orthophosphate, commonly present together in CAFO runoff, can be deposited from an alkaline solution with the addition of magnesium to form struvite ($MgNH_4PO_4 * 6H_2O$).[8] Struvite is known for its ability to be used as a slow release fertilizer.[8,11] Therefore, the successful removal of ammonium and orthophosphate, in the form of struvite, will aid in the reduction of eutrophication and provide a sustainable source of a phosphorus and nitrogen based fertilizer.

Available Technologies:

The issue of excess concentrations of dissolved nitrogen and phosphorus leading to eutrophication is well documented. Filtration techniques are being adopted or have already been put into place in many developed nations across the world. However, a new challenge faced by many wastewater sites regarding phosphorus removal is the capture of phosphorus in a useful form.[12] Listed below are multiple techniques featured in phosphorus removal.

Current-Day Phosphorus Removal

One of the most common forms of present day phosphorus removal is chemically dosing metal salts into water being treated to precipitate phosphorus from the wastewater.[12] These salts are added to the pre-treated influent of activated sludge, or the effluent stream before further purification. Aluminum and iron ions dissolved into solution form phosphate precipitates which can be removed from the solution through gravity or solid filtration.[13] The effluent stream for this process can have a phosphorus content below 0.3 ppm.[14] However, once the phosphorus is precipitated into a phosphate compound, it can be difficult to chemically separate the metals and phosphate.[15] Because the phosphates cannot be recovered, they have no economic use, therefore, this technique is only used to remove phosphorus, with no beneficial recovery or potential reuse.

One of the oldest techniques that continues to be utilized in wastewater treatment

facilities is the use of a naturally active adsorptive media for phosphorus removal. Naturally occurring minerals from wetlands are being utilized in treatment facilities. Dolomite has been noted as the most efficient source of phosphorus removal, removing 30-50% of phosphorus.[16] The onsite techniques act similar to wetlands, absorbing phosphorus before wastewater release, preventing downstream eutrophication.[17] This is a popular technology because the minerals used are relatively cheap and little to no human maintenance is required. However, an obvious downside is the inability to separate the phosphorus once it is absorbed. This technology therefore does not allow for the recovery and reuse of phosphorus.

Phosphorus Removal to Fertilizer Methods

Several newer and less utilized technologies were investigated to find the optimal process to remove phosphorus in the form of fertilizer. Enhanced Biological Phosphorus Removal (EBPR), hydroxyapatite crystallization (HAP), and nano-crystal methods are a few of the phosphorus removal techniques that were explored.

Enhanced Biological Phosphorus Removal (EBPR) is a process to remove phosphorus that is commonly used in wastewater treatment plants.[18] This process is composed of two major steps, an anaerobic stage followed by an aerobic stage.[19] Problems with EBPR for this application is the long aging period before entering the anaerobic stage, which would require a large containment pond, and the specific pH range required.[18] Furthermore, this technology focuses on the removal of phosphorus from sludge in wastewater, which is currently treated at CAFO facilities, rather than the dissolved phosphorus in the liquid water that is not treated.

Another experimental technology that has shown promise in removing phosphorus is through hydroxyapatite (HAP) crystallization ($Ca_5(PO_4)_3OH$) with cow bone as the seed material. HAP is a calcium phosphate similar to the hard tissues that exist in the human body.[20,21] The drawback of this method is that the calcination temperature needs to be above 600ºC for a reasonable phosphorus removal efficiency.[21] Secondly, the reaction is sensitive to the alkalinity of the wastewater solution and it is recommended that the cow bone go through a decarbonation process to decrease the sensitivity effects of alkalinity.[21] The combination of these highly specific conditions makes this technology labor and energy intensive.

The third alternative technology for phosphorus removal is using nano-crystals that take

advantage of phosphorus adsorption properties. The process involves placing crystals on the surface of the water where they sink to the bottom while catching phosphorus by adsorption.[22] A disadvantage of this method is that the phosphorus is recovered from the crystal through calcium precipitation, so further processing is required to retrieve phosphorus as a useful fertilizer.[23]

Proposed Technology

The selected technology will consist of the precipitation of orthophosphate and ammonium in the form of struvite in an electrochemical reactor that provides magnesium ions and an alkaline solution. Specifically, orthophosphate (PO_4^{3-}) in runoff can be reclaimed through the precipitation of struvite ($MgNH_4PO_4 * 6H_2O$) which can also serve as a phosphorus based fertilizer.[24,25] Struvite precipitation occurs in supersaturated conditions when Mg^{2+}, NH_4^{+} and PO_4^{3-} ions are present, described by the following stoichiometric equation:[26]

$$Mg^{2+}{}_{aq} + NH_4^{+}{}_{aq} + PO_4^{3-}{}_{aq} + 6H_2O_l \rightarrow MgNH_4PO_4 * 6H_2O_s$$
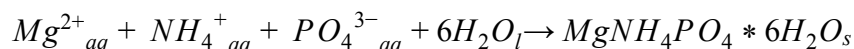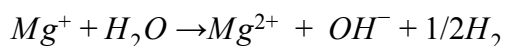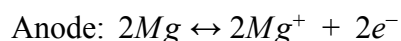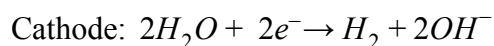
Formation is driven by high pH because orthophosphate is a triprotic acid that exists in its monoprotonated form (HPO_4^{2-}) in a neutral solution. Increasing alkalinity shifts the acid base equilibrium and removes the last proton from the orthophosphate, forming PO_4^{3-}. Only a small amount of orthophosphate in the form of PO_4^{3-} is required to begin struvite precipitation.[24] Supersaturation occurs in this alkaline solution, thus struvite precipitation is strongly dependent on pH and most closely follows first order reaction rate kinetics, which allows for the rate of formation to be predicted by the concentration of a single chemical species.[9,26] Alkaline conditions in a solution containing the required ions can be obtained by the addition of a base such as $NaOH$ or $Mg(OH)_2$, the stripping of dissolved CO_2 via air, or in the case of this proposed design, electrolysis of water or oxygen reduction to increase the pH at the cathode.[24, 25]

Optimal struvite purity has been noted to occur around a pH of 8.5 with the formation of impurities occuring at a pH greater than 10.5 in the form of magnesium phosphates or calcium phosphates, if calcium is present in solution.[25] This optimal pH is further supported by the fact that too high of a pH will cause the dissolved ammonium (NH_4^+) to be converted to ammonia (NH_3), which does not react and may be released from the solution as a gas. Agitation of the solution will also cause the removal of ammonia and thus additional ammonium must be added to the solution to aid in a higher struvite yield.[27] It has been proposed that the ideal molar ratio of

at least 1.1:1 ammonium to orthophosphate should be used.[27] Without an ideal ratio of ammonium ions in solution, other precipitates will form more rapidly. Both magnesium and calcium phosphates reduce struvite yields and are not considered useful fertilizers.

The application of electrical potential to the solution creates alkaline conditions to drive struvite formation without the addition of a basic chemical species. However, some base can be used in the initial solution to achieve a slightly basic starting pH before electrolysis is completed. Applied potential causes water electrolysis or oxygen reduction, both of which form hydroxide anions at the cathode.[25] The difference between these pathways occurs due to applied potential and rate limitations. The oxygen reduction requires a lower applied potential (-1300 mV/SSE) and is mass transport limited while water electrolysis requires a greater applied potential (-1700 mV/SSE) and is not limited by mass transport.[26,27] Despite potential cost savings with oxygen reduction, complications arise including the stripping of ammonia when oxygen is bubbled through the solution and reduced reaction rates due to mass transfer limitations. Some oxygen reduction will occur at the cathode until the oxygen has been consumed.

With hydroxide formation occurring at the cathode, typical electrolysis is paired with hydrogen evolution at the anode, assuming inert electrodes are used.[25] Addition of a source of magnesium to the solution is required in order to have all three ions present for struvite deposition. If a magnesium electrode is used as the anode, magnesium will be released into solution, giving the final electrochemical reactions for struvite formation as follows:[25]

$$\text{Cathode: } 2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

$$\text{Anode: } 2Mg \leftrightarrow 2Mg^+ + 2e^-$$

$$Mg^+ + H_2O \rightarrow Mg^{2+} + OH^- + 1/2H_2$$

The first step of magnesium cation formation is assumed to be the rate limiting step in its formation but has been noted to be increased by the presence of chlorine anions.[25]

With the use of a sacrificial magnesium anode in the presence of orthophosphate and ammonium (present together in CAFO runoff [5]), struvite can be formed. In the case of a pure orthophosphate solution as provided by WERC, ammonium must be added into the solution. In this case, NH_4Cl can be added in the preferential ratio of at least 1.1:1 with that of the orthophosphate. This will also account for added chloride ions which may accelerate the reaction

kinetics associated with magnesium cation formation, making orthophosphate the limiting reagent. Lastly, the basic species formed in these electrochemical reactions may aid in the sanitization of the wastewater being treated.[24]

Struvite precipitation has been often researched at orthophosphate concentrations of around 250 ppm (mg phosphorus/L) which is that of swine manure.[26] These concentrations far exceed natural wastewater runoff, found to be around 20 ppm, where phosphorus solubility is limited by that of calcium phosphates.[28] While research is lacking in the area of struvite formation from 20 ppm solutions of orthophosphate, it is hypothesized that the equilibrium formation of PO_4^{3-} from HPO_4^{2-} in a basic solution may have a significant rate reduction at low concentrations. Also, the reduced concentration of all reagents will affect the supersaturation level required for struvite formation. Lastly, because the reaction is proposed first order kinetics, the reactor size is directly dependent on concentration and low reactant concentrations would require a much larger reactor volume.[9] Combining these factors, it can be expected that an initial 20 ppm solution will negatively affect yields by slowing the rate of struvite formation as well as reaching an equilibrium before much of the phosphorus is removed from solution. Therefore, upstream concentrating of initial runoff via reverse osmosis may be beneficial to phosphorus removal before struvite precipitation is carried out.

Experimental Design:

Full-Scale Design and Implementation

The proposed full-scale design will begin with surface water collection from the CAFO waste treatment ponds. It has been found that these ponds typically hold around one year's worth of water and can cover upwards of 10 acres of land.[29] This design will process one year's worth of pond holdings in a one month time frame, thus drastically reducing pond size. Additionally, it is assumed that this process will only run for 90% of the time to account for general maintenance and possible repair. This will call for a continuous processing feed rate of 2.6 m^3/hr assuming an inlet flow rate of 53.2 m^3/day coming from the facility.[29] The solid waste present in the pond will settle to the bottom forming a layer of sludge. This technology will focus on the dissolved orthophosphate in the liquid layer on top, found to be at a concentration of 20 ppm.[28] Removal of dissolved orthophosphate and ammonia within the pond wastewater will occur as a continuous

process in three phases. These include Phase One, upstream filtration and concentration of the orthophosphate via reverse osmosis (RO), Phase Two, electrochemical deposition of struvite in an EPFR, and Phase Three, downstream separation of struvite particles from solution and drying. This can be seen in the process flow diagram of this proposed process provided in Figure 1.
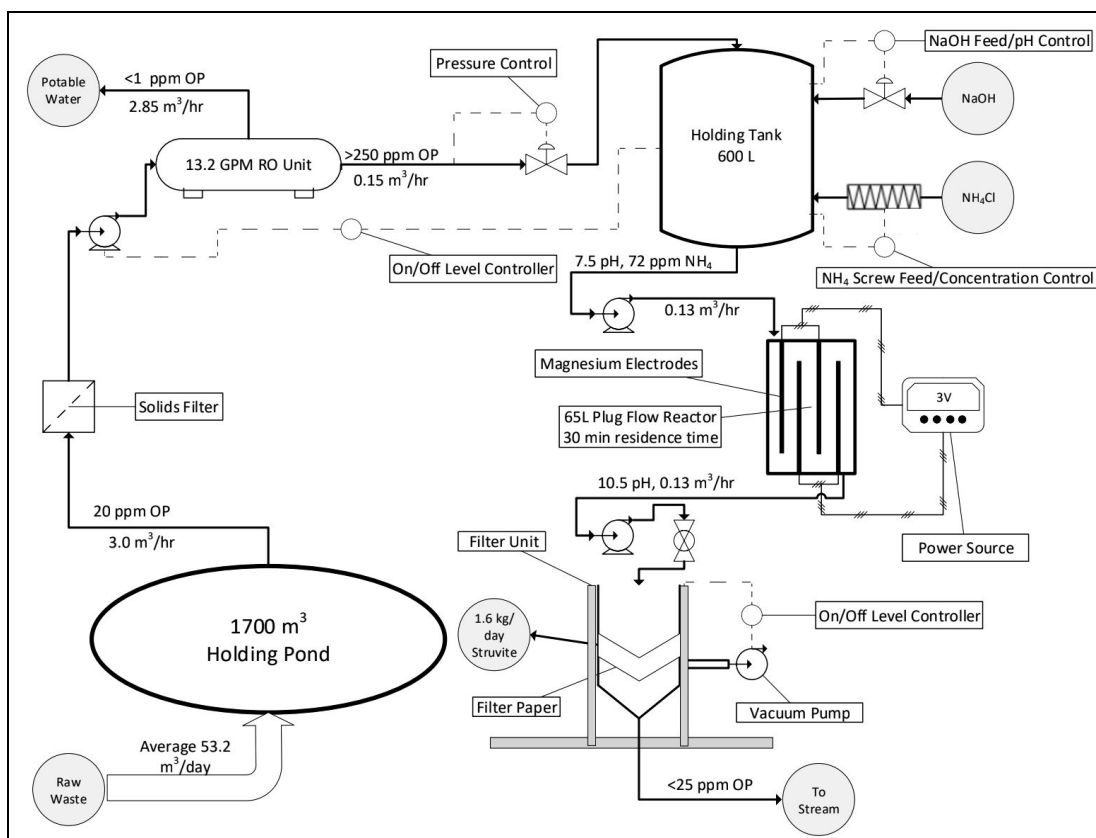

Figure 1. A process flow diagram of the proposed full-scale design

Phase One will require filtration of solids from the aqueous phase such as particulates of dirt, algae, waste products, and others that may cause a blockage in the RO membrane. It is suggested that this filtration unit consist of a membrane to remove matter down to 0.45 μm, based on previous work done on CAFO runoff purification,[25] assuming most of the solids have settled out within the lagoon. The reverse osmosis stage serves to concentrate the initial solution with 20 ppm orthophosphate to the desirable range of 200 to 300 ppm before it is fed into the reactor. Reverse osmosis utilizes pressure created by a pump on the concentrate side of the unit which forces pure water through the membrane. Pressure is adjusted by a downstream valve on the concentrate side. This will leave behind salts and impurities on the concentrate side. A feed

rate of 13.2 gpm (3 $\frac{m^3}{hr}$) will be processed by the full-scale RO system in order to stay ahead of the reactor processing rate. The reactor will thus be the rate limiting step. The volumetric flow rate of the RO feed will be reduced to 1/20th, 0.66 gpm (0.15 $\frac{m^3}{hr}$), creating the ideal 200-300 ppm solution, assuming some passage of ions through the membrane.

After reverse osmosis, the concentrated orthophosphate solution will be stored in an 160 gal (600 L) holding tank which allows a steady supply of feed to the reactor. Ammonium chloride may be added if the wastewater source does not have an excess of ammonium present. Sodium hydroxide will be added to achieve a pH of 7.5. The pH and ammonium content in the holding tank will be monitored and adjusted by controllers. The solution in the holding tank will be fed to a 65 L EPFR with magnesium electrodes. The electrodes in the EPFR will be staggered with the anodes attached to one wall extending across the reactor nearly to the other wall, and the cathodes attached to the opposite wall extending in the same way. These electrodes will act as baffles that direct the flow, effectively increasing the exposed surface area of the electrodes. The EPFR will have a residence time of 30 minutes, resulting in a required flow rate through the reactor of 0.57 gpm (0.13 $\frac{m^3}{hr}$) in order to meet the processing requirements of 1700 m³ in 30 days. The reactor is expected to achieve a 90% conversion of orthophosphate to struvite.

A power source connected to the reactor will provide a continuous 3V of potential spread across 1.43 m² of magnesium electrode surface area. The reactor will be a rectangular vessel with a removable lid. The dimensions will be 1.9 m x 0.4 m x 0.4 m. The reactor effluent stream containing solid particulate struvite will be allowed to vacuum filter through filter paper. The filter paper will collect a wet struvite cake for an approximate total of 1.6 $\frac{kg}{day}$ which can be regularly emptied and the filter paper replaced. It is expected that all equipment for this entire process can be placed on a 20 m² cement pad near the wastewater pond.

The struvite particulates being collected in the filter paper will require a planned maintenance time. It is expected that changing the filter paper will require halting the reactor flow for approximately 15 minutes and turning off the power source. Once the solid is collected, struvite can be stored dry or damp providing that it is not exposed to an acid solution. It is also expected that the electrodes may require occasional power washing to remove any struvite build up that reduces performance. This will require about one hour of time on a weekly basis by an

operator. Along with power washing, some solid build up will occur along the bottom of the reactor where particles are too heavy to be carried via flow. The washing should also act to push these solids to the exiting pipe. Lastly, as discussed later in the limitations section, the RO membranes and sacrificial magnesium electrodes will require replacement which has not been addressed in this analysis, and their predicted lifespans are unknown. The electrodes proposed here are all made of magnesium, however, further investigation may show that other inert materials can be used at the cathode or only part of the anode be made of magnesium to reduce excess usage of magnesium when not necessary. The sizings for this process can be implemented into a large sized CAFO facility and therefore, the process would require resizing for smaller facilities and other applications.

Bench-Scale Design and Testing

The development of the bench-scale design involved multiple approaches working toward the objective of maximizing orthophosphate recovery and struvite purity within a reasonable processing time. The final design utilizes an EPFR with magnesium electrodes.

Reverse Osmosis

Reverse osmosis (RO) is used to concentrate the 20L of 20 ppm orthophosphate solution to 1L at approximately 250-270 ppm. The process of reverse osmosis requires pressure to be applied in order to push water through the membrane while impurities remain on the opposite side of the membrane.[30] The pressure in this design is applied by a feed pump and a ball valve located on the concentrate stream. The valve can be positioned to adjust pressure on the membrane, which is read by a pressure gauge. The bench scale design utilizes a pressure of 60 psi. Approximately 35 minutes are needed to concentrate 20L of 20 ppm orthophosphate solution to 1L. After the solution has been concentrated, it can be pumped into the reactor.

Reactor Studies

Bench-test 1, consisting of initial experimentation with struvite precipitation from 20 ppm and 250 ppm orthophosphate solution via the addition of sodium hydroxide to create a basic solution. This batch experiment was performed for one hour. Magnesium chloride was added to the solution in a 1:1 ratio with dihydrogen ammonium phosphate. Filtered solids showed 50% and 57% orthophosphate recovery in the form of struvite was observed from the 20 ppm and 250

ppm solutions, respectively. It was observed that struvite formation in the 250 ppm solution occurred quicker than in the 20 ppm solution by monitoring when visible precipitates formed. While this method successfully produced struvite, constant addition of base to drive the reaction is not economically feasible in full-scale implementation.

Water electrolysis was then used to increase the pH around the cathode, causing struvite to precipitate from the solution. Bench-tests 2, 3 and 4 consisted of electrolysis performed in a beaker using nickel electrodes at 15 V of applied potential. However, these experiments produced a green solid, most likely nickel hydroxide. It was concluded that the resistance in the system was too great to allow water electrolysis to occur before nickel dissolution. This was a direct result of the intrinsic properties of the nickel metal, passivation of the nickel, and the large distance between the electrodes. The corrective action decided upon was to decrease the distance between electrodes and change electrode material.
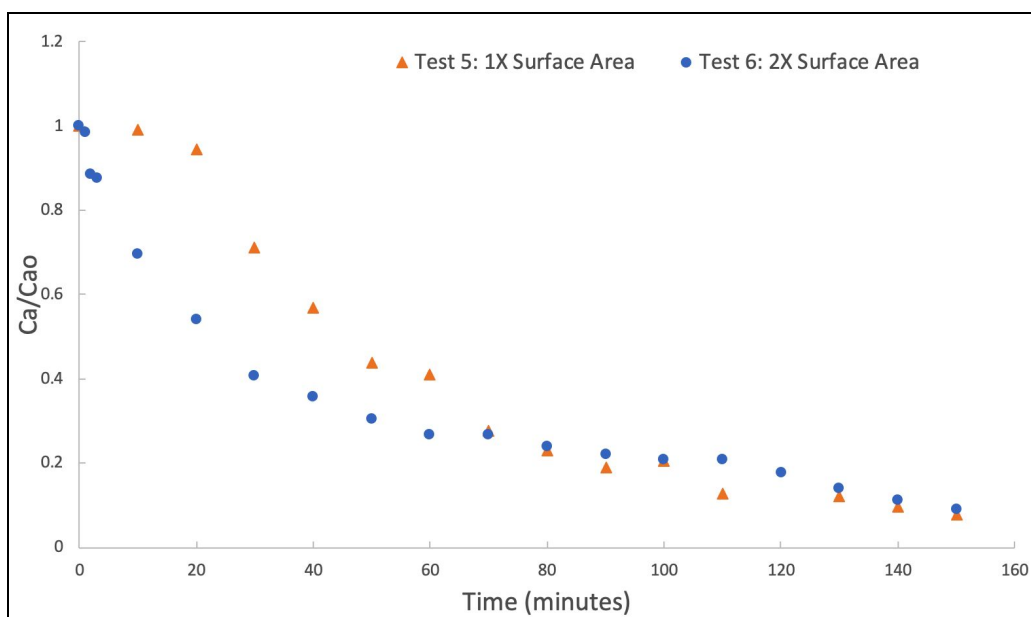

Figure 2. Orthophosphate concentration over time for batch electrolysis

Further experimentation in bench-test 5 was performed in a batch system using sacrificial magnesium electrodes with the dimensions of 4" x 2.5" for a total electrode surface area (SA) of 40 in^2 in a solution of 250 ppm orthophosphate. The applied current causes magnesium dissolution, eliminating the need to add magnesium as a salt. A potential of 5 V was applied until the bulk solution pH reached 10.5, the upper range at which struvite purity begins to decrease,[25]

and then the voltage was reduced to 3 V to keep the pH between 9-10. The solution became visibly cloudy around 40 minutes, but the experiment was continued for a total of 150 minutes. A curve of orthophosphate concentration over time is shown in Figure 2. The y-axis is shown as the concentration over the initial concentration to allow comparison to other trials despite small differences between initial and final concentrations.

From Figure 2, it can be seen that the precipitation of struvite did not begin until about 10 minutes with the smaller surface area trial. This is due to the initial orthophosphate and water solution being slightly acidic and time was required to increase the pH of the solution through electrolysis. To eliminate the time delay at the beginning of the experiment, the initial solution was pretreated with sodium hydroxide to increase the starting pH to 8.5. In addition, the SA of the electrodes was doubled for a total of 80 in^2 in bench-test 6 also shown in Figure 2. This curve shows that the reaction begins immediately. Upon comparison, excluding the initial delay in the 1X SA, doubling electrode area caused minor increases in reaction rate, however, both gave the same final orthophosphate conversion of only about 80%. Bench-test 6 conditions were replicated in bench-test 7 with a similar outcome. Since greater conversion could not be achieved after more than two hours of reaction time and the batch system had limitations on the maximum surface area that could be obtained for greater conversion, a plug flow arrangement was tested.

The next experimental approach, in bench-test 8, utilized a 1.6 L EPFR, with a total electrode surface area of 144 in^2 and achieved a 97% conversion of phosphorus when processing 5L of 250 ppm of solution. This reactor was larger than required for the bench scale and a new EPFR was built with a 0.5L volume and electrode surface area of 96 in^2 to keep the SA to volume ratio the same as the first EPFR. A 250 ppm orthophosphate solution at a pH of 8.5 was pumped through the reactor at about 20 mL/min, resulting in a residence time of 25-30 minutes in bench-test 9. A potential of 3 V was applied to the electrodes because a larger area allowed for a lower voltage to achieve the same pH. The configuration of the EPFR can be seen in Image 1.

The EPFR reached steady state in one residence time, however, the exiting pH reached 10.7 when the initial solution was adjusted to 8.5. Because of this, the initial pH adjustment was only made to 7.5 in bench-test 10, causing the exit stream to remain below 10.5, but did not affect overall conversion of phosphorus. Both trials resulted in single pass orthophosphate

conversion of about 93% once at steady state. The concentration profile of orthophosphate over reactor length is shown in Figure 3. This type of EPFR was chosen for use in the final design.

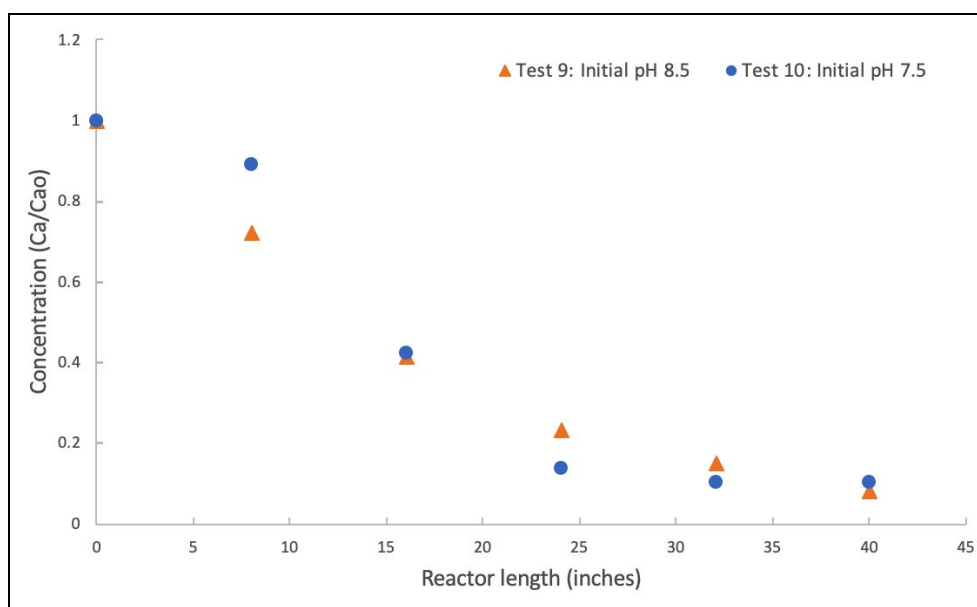Image 1. Top down view of the bench-scale continuous electrochemical plug flow reactor

Figure 3. Orthophosphate concentration over total length traveled in a continuous EPFR

Filtration and Solid Identification

The solid struvite particles were removed from the solution using a vacuum flask filtration system. The mass of the filter paper was recorded before use and after filtration with the collected solid to determine the mass of water and solids. The filter paper was left out to dry for nearly 24 hours before weighing to ensure that water was not included in the mass of solids. By pulling vacuum through the sample for 10 minutes, less than 25% water remained by mass. Solids were analyzed via X-ray diffraction to confirm the presence of struvite. Struvite was positively identified in the batch and EPFR samples while one EPFR sample showed some

Cattiite, which is a magnesium phosphate precipitate. This impurity was thought to be a result of allowing pH to rise too high (above 10.5) and was be corrected for by lowering operating pH.
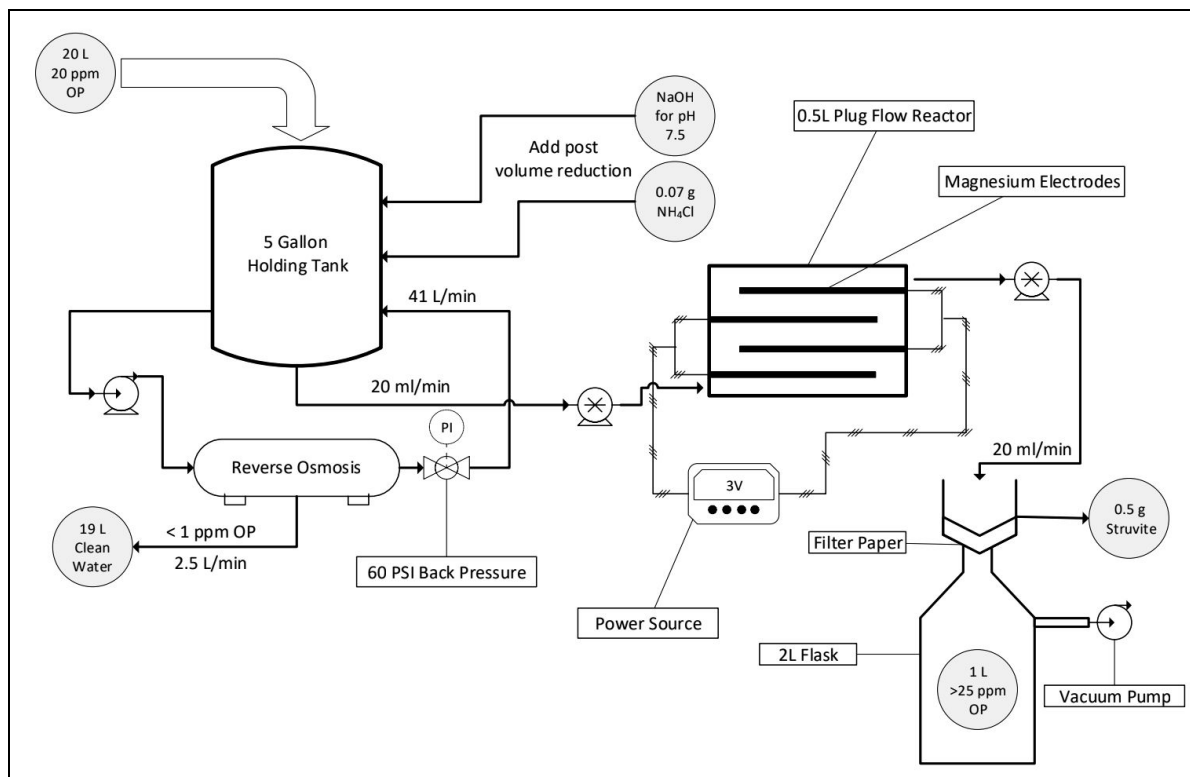
Complete Bench-Scale Design

Figure 4. A process flow diagram of the proposed bench-scale design

The proposed complete bench-scale process is shown in Figure 4. The five gallon holding tank will consist of a 5 gallon bucket which will contain the initial 20L of 20 ppm solution provided by WERC. It is projected that the total time to run this process will be about 2.5 hours. A centrifugal pump will be used to complete the reverse osmosis while two peristaltic pumps are employed around the reactor. Finally, vacuum filtration will be used to collect struvite.

Health and Safety:

Full-Scale Operation

Hazards such as chemical and electrical exposure affecting the health and safety of the CAFO site, operators, and surrounding water sources are implemented within a CAFO facility's written Hazard Communication Program (HCP). The HCP will be in compliance with personal protection and environment safety standards dictated by the Occupational Safety and Health Administration (OSHA) and state and federal Environmental Protection Agency (EPA). To

ensure safety in the workplace, safety data sheets (SDS) for ammonium chloride, magnesium, sodium hydroxide, and struvite, will be readily available to all who regularly handle, store, and utilize the chemicals in accordance with OSHA.[31-34] In case of emergency, local fire departments and emergency response personnel will be issued copies of the SDSs and the HCP.

The concentration of orthophosphate in the effluent stream is designed to be monitored remotely, however, struvite collection and system maintenance will be done manually. Prior to struvite collection or system maintenance, the main power source must be shut down. A Class D fire extinguisher and multimeter should be readily available to prevent injury or fire from an electric source. Operators must be appropriately clothed in the protective eyewear, gloves, and a dust mask to reduce acute exposures to ammonium chloride, struvite, and sodium hydroxide that upon contact cause skin, eye, and respiratory irritation. Loss of containment from the holding tank will be mitigated through a level controller that stops and starts the pump controlling the flow rate through the RO unit. An interlock will automatically cut power to the pump if the level exceeds 90% of the tank volume. Any unintended power surges will result in power to the system being shut off such as that of a short circuit. The reactor will be open to the atmosphere to prevent risk of hydrogen accumulation produced by the electrodes.

For the safety of the general public and surrounding aquatic life, all records pertaining to the release of effluent streams into nearby waterways will be maintained until the EPA terminates the National Pollutant Discharge Elimination System (NPDES) permit provided to the CAFO facility. The HCP will be reevaluated annually to ensure it meets its safety objectives and will be revised when appropriate to address any changes to equipment or process. The EPA will conduct periodic inspections under Section 308 of the Clean Water Act to review, copy records, and collect samples or other information from effluent stream entering surrounding water sources.[35]

Bench-Scale Operation

In addition to the health and safety information and precautions stated for the full scale design, proper laboratory attire is required while operating the bench-scale system. Although operating at a very low voltage, exposed wires near the solution can cause electrical shock if touched. If a fire were to occur, the power source should be turned off to ensure the safety of the

area. Hydrogen is produced in small quantities by the electrodes. The reactor is open to the atmosphere to allow for the dispersion of the created hydrogen, negating any risk of pressure build up and flammability hazards. The bench-scale operation will be equipped with a larger secondary containment vessel in case of a spill or leak. A circuit-breaker-protected outlet strip will be also be used to prevent injury, fire, or damage to the equipment due to an electrical short.

Legal and Regulatory Issues:

North Carolina abides by federal and state regulations to ensure runoff effluent meets all standards put in place by The North Carolina Department of Environmental Quality (NC-DEQ), North Carolina Department of Environment and Natural Resources (NCDENR) and the United States Environmental Protection Agency (EPA).

The management and education behind acceptable effluent nutrient concentrations is a newer and less investigated field. This limitation has resulted in few regulations and penalties for phosphorus and nitrogen rich runoff streams. Research from the EPA has presented consequences for these streams specific to North Carolina and has been working with environmental agencies in the state to create nutrient concentration management laws and penalties for malpractice. The NCDENR-Division of Water Quality ensures that all nutrients found within runoff streams in the state abide by all rules and regulations found in Section 0.0200- Classifications and water quality standards applicable to surface waters and wetlands of North Carolina. These impurities are detailed in Appendix A of the North Carolina Administrative Code 15A.[5] While this document does not give insight to the best practice concentrations of effluent streams applicable to phosphorus or nitrogen, studies have found that there are concentrations safe to emit within runoff streams which will not promote eutrophication. Algal productivity was shown to be moderately low to very low for concentrations of 0.2-0.4 and 0.005-0.010 ppm for nitrogen and phosphorus, respectively. However, algal production increased from moderately high to very high categorization when concentrations exceeded 0.3-1.5 ppm of nitrogen and greater than 0.10 ppm phosphorus.[36] It can be concluded that concentrations exceeding these values in North Carolina Runoff streams are hazardous to the environment and induce damage to waterways.

North Carolina currently has no numerical regulations for total nitrogen and phosphorus

in any type of waterway.[37] As a preventative measure for the affected streams in North Carolina, the NC-DEQ has provided regulations and permits regarding CAFO facilities; the main source for over-concentration of nutrients in runoff streams. Facilities must attain and maintain all permits and regulated operation to follow standards implemented in the North Carolina General Statutes regarding Animal Waste Management Systems.[38] It is assumed that CAFO facilities maintain all mandatory permits for the daily operation before design implementation.

The federal EPA's Clean Water Act provides the framework for regulation of discharge of pollutants into waterways within the United States.[39] CAFOs are considered large, medium or small facilities, defined by the number of animals present. Each size has mandatory permit and plan requirements associated with discharge pollution. All large CAFOs are required to obtain a NPDES permit and submit a Nutrient Management Plan (NMP) in order to operate and must be maintained until the CAFO is properly closed.[40] The NPDES permit sets nutrient effluent limitations based on technology available to control the pollutants that are economically achievable for a CAFO. Regulations for technology-based effluent for CAFOs are defined by Federal Regulation 40 CFR part 412.[41] Medium and small CAFOs are not mandated to obtain permits. However, these facilities are subjected to the same effluent guidelines followed by large CAFOs and must use best professional judgement (BPJ) for any pollution discharged.[40] Guidelines for swine specific CAFOs are stated in Sections 2.2.2.2-2.2.2.7 in the NPDES Regulation and the Effluent Guidelines for Concentrated Animal Feeding Operations.[40] Permits mandated for CAFO facilities are differentiated by NPDES types. There are two types of NPDES permits: general and individual. A general permit covers facilities with similar qualities within the same geographical region. An individual permit is specifically tailored to an individual facility, so waste and discharge management systems must be evaluated on a case-by-case basis. An individual permit is only valid for a maximum of five years.[42] CAFO facilities can obtain either of these two permits, depending on their geographical location and operation.

A Nutrient Management Plan (NMP) addresses the risk of nutrient runoff and defines a nutrient conservation and management plan for a facility. Requirements for a NMP are defined by NRCS Conservation Practice Standard Code 590.[43] However, states have the authority to implement more stringent requirements. For example, North Carolina assesses the risk of

phosphorus loss from the facility with a Phosphorus Loss Assessment Tool (PLAT) test. This test must be done at least once every five years.[44] Utilization of this design will be implemented into a CAFOs nutrient management plan to aid in meeting regulation requirements.

Lagoons, are a notable aspect for CAFO facilities. Lagoons are defined as a necessary pond or basin utilized to receive, hold, and treat wastewater for a period of time.[45] A common lagoon heavily used in wastewater treatment at CAFOs is an anaerobic lagoon.[46] Anaerobic lagoons degrade organic animal waste into carbon dioxide and methane. Lagoons are a required component to mitigate pollution in CAFO facilities and are used to prevent effluent from directly entering waterways.[40] Placement and overall lagoon design hold strict guidelines and are detailed in the NRCS, Conservation Practice Standard - Waste Treatment Lagoon (Code 359).[47] Maintenance and routine inspections of these ponds are necessary multiple times a year. These guidelines are summarized by the EPA-CWA and are supported by the state of North Carolina's need for NPDES Permits. These necessary permits are mandated by the NCDEQ in accordance to the North Carolina General Statutes.[48]

Economic Analysis:

Economic Justification of Process

The implementation of this technology is attractive from an economic standpoint for a few key reasons. While regulations on the amount of phosphorus in the waterways is not currently enforced, it is anticipated that policies will soon become more stringent, thus limiting the amount of livestock a given CAFO can handle. This process will lower the phosphorus concentration in the runoff and allow an increase in the amount of livestock that can be housed. As the CAFO increases its number of livestock, they will also increase in profit. CAFO facilities utilizing this technology could also claim their products as "sustainably raised" which may increase profitability as meat producers and consumers move more to organic and sustainably raised livestock. In addition, potable water that will be produced through reverse osmosis and the effluent coming from the reactor can be stored and reused around the CAFO facility, saving money on utilities over time. This offers an economic incentive, as the cost to produce 1000 gallons of water using the proposed method around 2 cents, whereas the average cost of 1000 gallons of water in one of North Carolina's most CAFO dense regions, Sampson County, is

$4.85 .[54] Lastly, precipitation of struvite from wastewater provides a renewable source of a phosphorus based fertilizer, which is increasingly important as the natural supply of phosphate rock becomes limited over time.

Struvite, a slow-release fertilizer, is best used for long term phosphorus deposition into soil. Struvite is nearly insoluble in water, therefore preventing nutrient pollution and eutrophication from runoff, making it an environmentally safe fertilizer.[49,50] Struvite has been found to have a global market price of $530/ton.[51,52] The proposed design will produce approximately 584 kg/yr of struvite, equating to a possible $310.25/yr in struvite sales. This shows that struvite is not economically viable for commercial production at this scale. Therefore, it is recommended that owners of this equipment use struvite for their own fields to reduce their own operating costs or sell it to neighboring farms or agricultural centers. It is also recommended that wastewater treatment plants consider this technology. Phosphorus recovery as struvite acts as a preventative measure in wastewater treatment sites by removing nutrients that cause precipitation buildup in piping and equipment.[52] This practice is implemented in one treatment site reporting saving $450,000 per year.[53]

Given that the primary objective of the system is to reduce phosphorus concentration in the outlet stream, the struvite collection may be viewed as an additional benefit. Although production of struvite for solely commercial sales may not be economically viable, this proposed design offers CAFO facilities an economic and simple solution to meet increasingly stringent environmental regulations.

Capital & Operating Costs

The predicted operating and installation costs of this process are shown in Table 1. These calculations are based on a design that processes 53.2 m^3 of wastewater a day. Costs may vary depending on the scale of the system. Table 2 shows a breakdown of the cost of individual pieces of equipment. Since there are no harsh chemicals or operating conditions involved in this process, plastic tanks will be purchased for the reactor and holding tank.

Table 1. Major operating costs and investments, including a total cost of manufacturing

Fixed Capital Investment ($)		21,000
Operating Costs	Labor ($/yr)	3,650
	Raw Materials ($/yr)	4,000
	Utilities ($/yr)	80
Cost of Manufacturing ($/yr)		21,000

Table 2. A unit cost and quantity breakdown considering each individual unit operation

Unit	Quantity	Cost ($)	Unit	Quantity	Cost ($)
Reactor Tank	1	$230	RO Pump	1	$326
Holding Tank	1	$354	Tank/Reactor Pump	2	$130
RO Unit	1	$8,000	Power Source	1	$40
Magnesium Electrodes	3	$309	RO Membrane	4	$772
Total Cost ($)		$10,161			

The fixed capital investment is the summation of the purchase prices of the equipment with factored in installation costs. The labor costs are for one operator working 30 minutes per day. The raw materials account for the cost of NaOH, NH_4Cl, replacement of the sacrificial anode about once a month, and RO membrane replacement every two years. The utilities account for the cost in electricity to run the system. The cost of manufacturing is the cost of running the proposed design for a year. The total cost of each individual unit adds to $10,161 without including installation costs. The installation costs for each unit is included in the cost of manufacturing. Utilizing the straight line depreciation method, the annual value of depreciation over the course of nine years equates to $22,044, which is approximately the cost of the initial installation.

Limitations:

The proposed design and technology contains limitations, mostly in the form of knowledge gaps surrounding the technology, which must be addressed before wide-scale implementation can occur. The first is that the concentration of ammonium present at individual facilities is unknown. If ammonium is not present in the required concentrations, it must be purchased and fed to the reactor, which is an additional operating cost. Preliminary research shows that ammonium will already be present at amounts equal to that of orthophosphate and more will be added for an excess of 1.5:1 ammonium to orthophosphate which increases yields.[29] Next, the magnesium electrodes have not been tested for long term performance and overall lifespan. It is predicted that over time, magnesium oxide, struvite, and other precipitates may build up. Although they will be cleaned periodically, pitting will still occur as the metal is corroded away and used in the reaction. This will reduce the available exposed surface area, slowing reaction rates. Ultimately, this lifespan depends directly on the purity of the electrodes. The final effluent pH is currently predicted to be around 10.5 and this may need neutralized before release. Lastly, the proposed reactor has not been optimized in all respects. Magnesium concentrations in the effluent stream have not been tested and it may be desirable to make the cathode and partial anode materials out of inert materials to reduce magnesium waste. Also, the addition of calcium in the full-scale system may reduce struvite recovery by forming calcium phosphates. This most likely will need addressed by adjusting the pH of operation.

Minor limitations exist in terms of the economics of this technology. Additional research is required to determine projected costs of startup of the proposed operation, including the facilities for manufacturing and hiring of representatives to advertise and sell the technology. Market research is also required to determine the percentage of CAFO owners who are environmentally conscious and would willingly implement this technology for the betterment of their surrounding ecosystems.

Conclusion:

The proposed orthophosphate recovery process consisting of a reverse osmosis unit, an EPFR, and a solids filter to produce and collect struvite reduces nutrient pollution, produces a

useful fertilizer, and increases usable land area at CAFO facilities. It also accomplishes all tasks set forth by WERC. This technology provides an environmentally conscious solution that addresses concerns for water quality relevant to swine CAFOs and paves the path for nutrient management regulation across North Carolina and ultimately the United States.

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To: Ohio University, Task 5

From: Jeffrey C. Messaros

Date: March 1, 2019

Re: Legal and Regulatory Review

I have completed the Legal and Regulatory review for the Ohio University paper for the Removal and Reuse of Phosphorus as Fertilizer from CAFO Runoff. The Ohio University team did an excellent job at covering applicable environmental regulations.

The team covered appropriate federal and state regulations. The Clean Water Act, National Pollutant Discharge Elimination System (NPDES) permit, and North Carolina specific regulations were covered in significant detail during their evaluation. I have no substantial recommendations for changes within their paper.

I recommend several administrative changes to the paper. The National Pollutant Discharge Elimination System (NPDES) permit is first mentioned on page 17 and NPDES should be spelled out initially on that page. On page 18 EPA is spelled out but is previously defined on prior pages.

The paper states that regulations for technology-based effluent for CAFOs are defined by Federal Regulation 40 CFR part 12. I believe the regulation is actually 40 CFR Part 412.

As the Site Manager at the Naval Reactors Facility for BWSR, LLC I have approximately 20 years of experience in working directly with federal environmental regulations.

Again, the Ohio University team did an excellent job at covering the environmental regulations in significant detail.

Please let me know if you have any questions or need any additional information.

Sincerely,

Jeffrey C. Messaros
Site Manager – Naval Reactors Facility
BWSR, LLC

American Electric Power
1 Riverside Plaza
Columbus, OH 43215
aep.com

Eric J. Wittine
Director - Customer Solutions Development
AEP Service Corporation

614-716-2210

March 6, 2019

Dear Sir or Madam,

Attached to this letter are my audit findings related to the economic analysis section of the paper I was asked to review for the Ohio University WERC team. In general, I found the paper to be thorough, well written, and analytically sound.

Since graduating with a B.S.ChE in 2002, I have been employed in various engineering and technical roles within the electric utility industry. In these roles, I have performed various project and process engineering duties at coal and gas fired power plants. In addition, I have been involved in the design and permitting processes for a number of power plants, and have supported regulatory filings at the state and federal level. In my current position, I design and assess business cases for customer-focused product offerings related to customer-sited generation, electric vehicle charging, and energy advisory services.

Best Regards,

Eric Wittine

Enclosures – Task 5 Audit Notes

Task 5 Audit Notes (Economic Analysis Section):

• Certainly environmental regulations tend to become more stringent over time, but supporting the economics based on anticipated regulation could use a citation. In the absence of regulation, the ability to market livestock as "sustainably raised" *may* increase the value of the product and provide some incremental offset to system costs.
• The total cost breakdown discussion would benefit from showing the unit costs and quantities of each input. This goes for capital equipment, as well as, operating expenses for items such as labor and individual consumables. Without those, the reader is left to take the estimate at face value.
• Estimate items such as overheads and depreciation expense appear to be absent.
• The lack of unit costs and quantities also makes it difficult to determine if the utility expenses are reasonable. An annual electricity consumption estimate and unit costs for electricity would be necessary precursors to the stated cents/1000 gallons value.
• Try to annualize all estimate numbers to allow for apples to apples comparisons. The $0.85/day struvite price and $4/second water savings estimates are examples.

OHIO
UNIVERSITY

Risk Management and Safety

University Service Center   Phone (740) 593-1666

Athens, OH 45701 - 2979   Fax (740) 593-0808

DATE: March 4, 2019

TO: Dr. Darren Ridgeway, ChBE Department; WERC Student Design Team

FROM: Nancy Rauschenberg, Laboratory Safety Compliance Officer

SUBJECT: Review of Task 5 Project Design Health and Safety

Dr. Ridgeway,

Your student team should be congratulated on their creative solution to this design challenge! Your design combines some standard elements with an unexpected twist, utilizing electrochemistry to accelerate an otherwise slow reaction to practical scale.
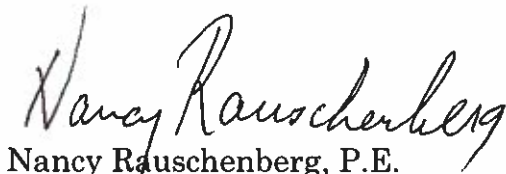
I have the following specific suggestions that your team should consider.

1. For your bench-scale process, please be sure to include containment in case something spills, a hose slips out of a vessel, etc.

2. Be sure to have a 3-ring binder or similar, with printed SDS of all your chemical materials, whenever you have the benchtop unit set up for demonstration. Operators should have suitable PPE for their own use, such as safety goggles, gloves and lab coats, guided by the SDS recommendations.

3. I suggest using a circuit-breaker-protected outlet strip to protect your demo unit in case some of your liquids spill or any other situation arises that might short-circuit the power to your pumps.

4. In the written project report draft, the electrochemical reactor is repeatedly referred to as a "plug flow reactor" and this is incorrect. The term "electrochemical reactor" is correct for the unit operation in your design.

5. For your full-scale design concept, I would suggest looking for continuous filtration technology that could handle the wetcake that you are collecting, rather than filter paper. At some point, economies of scale will kick in and you will want to be able to model running continuously.

As a chemical engineer with 35 years of experience doing manufacturing process design and development for several Fortune 500 industrial corporations, I am well-qualified to review your project. I am registered as a Professional Engineer in the State of Ohio and currently employed as a Laboratory Safety Compliance Officer here at Ohio University.

Best of luck with the design competition!

Nancy Rauschenberg, P.E.
Laboratory Safety Compliance Officer
Ohio University

Legal and Regulatory - Site Manager, Naval Reactors Facility - Jeffery C. Messaros

Remarks from Auditor:
1.) Properly address acronyms for EPA and NPDES and define them before using them.
2.) Update the federal regulation for technology-based CAFOs.

Comments Addressed:
1.) Updated acronyms.
2.) Matched the correct section the federal regulation.

Economic Justification - AEP - Eric Wittine

Remarks from Auditor:
1.) Annualize all estimate numbers for apples to apples comparisons.
2.) Provide a citation or provide an alternative to a citation to increase possible profitability.
3.) Provide an unit cost and quantities breakdown.
4.) Depreciation not mentioned.

Comments
1.) Updated the number for units in $/yr.
2.) Livestock products from CAFOs utilizing this technology could also claim their products as "sustainably raised" which might increase profitability as produce and meat products move more to organic and sustainably raised livestock.
3.) Created a unit cost breakdown table.
4.) Depreciation has now been mentioned.

Health and Safety - Laboratory Safety Compliance Officer - Nancy Rauschenberg

Remarks from auditor:
1.) Have a 3 ring binder available with all the SDS sheets available and all operators of equipment should have the PPE as guided by SDS.
2.) Be sure to include some type of containment for spills and leaks.
3.) The reactor should be called an electrochemical reactor and not a plug flow reactor.
4.) To use a circuit- breaker protected outlet strip to protect our demo unit in case some of our liquids spill or any other situation that arises that might short-circuit power to pumps.
5.) Look for continuous filtration technology that can handle a wet-cake to collect the final struvite rather than filter paper.

Comments addressed:
1.) This was already stated in the full scale operation, and it was stated that the same guidelines apply for the bench scale to save typing room. A safety binder including relevant Safety Data Sheets and experimental safety plan provided to WERC was also generated and will be readily available when bench operation is in progress.
2.) Stated "The bench-scale operation will be equipped with a larger secondary containment vessel in case of a spill or leak." in the health and safety section
3.) PFR is changed to EPFR to stand for electrochemical plug flow reactor
4.) Included this in bench scale design and packing list
5.) Alternative continuous filtration methods were originally considered. However, after considering the amount of struvite produced from full scale operation (1.6 kg/day), it was decided that this amount of solid did not constitute the need for a continuous filtration system to be used. Therefore the filtration system was kept as initially designed.