

# Electrochemical Wastewater Treatment: Nutrient Recovery and Selective Contaminant Removal

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## Abstract

Electrochemical approaches and technologies have the potential to be used and applied to a wide range of water and wastewater treatment needs, particularly if these technologies are paired with a renewable energy source such as solar energy. Renewable-powered electrochemical technology can be used for rural, remote applications and in other scenarios where non-chemical approaches are desirable. Electrochemical technologies can also be potentially used where electrode and electrocatalyst design can be tailored to address specific treatment requirements, such as selective contaminant removal, resource recovery, and disinfection. The research results presented here focus on two applications of electrochemical technology to wastewater treatment: electrochemical precipitation of magnesium ammonium phosphate, known as struvite, for nutrient recovery and simultaneous ammonia oxidation and disinfectant generation for aquaculture wastewater. Experiments on electrochemical struvite precipitation comparing a pure magnesium and a magnesium alloy electrode demonstrated that the pure magnesium electrode produces more current; both anodes result in a crystalline struvite precipitate, as confirmed by x-ray diffraction. Cyclic voltammetry studies for aquaculture wastewater treatment using graphite and graphite-supported PtRu films as the anode demonstrate that the presence of PtRu reduces the voltage required for ammonia oxidation. When graphite is used as the cathode, the disinfectant molecule hydrogen peroxide is produced, suggesting electrochemical disinfection is possible in parallel with ammonia oxidation.

*Keywords: Electrochemical Water Treatment, Struvite, Ammonia Oxidation, Disinfection, Electrode Design*

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## 1. Introduction

The use of electrochemical processes and technology has been a focus for the energy conversion and storage fields, as technologies ranging from batteries to fuel cells and electrolyzers have received significant attention as cutting-edge and advanced next-generation technologies. To date, the application and development of electrochemical technologies for water and wastewater treatment needs have been limited to specific niches; for example, electrochemical technology has been a focus for non-membrane-based desalination (e.g., electrodialysis or capacitive deionization) and for electrocoagulation of metals and organics, as a replacement for chemically-driven coagulation. However, electrochemical processes and technologies offer a much greater range of possibilities for wastewater treatment needs and goals. From chemical-free disinfection to electrocatalytic conversion of contaminants to recovery of critical nutrients, electrode engineering, control of operational voltage and current windows, and optimization of device design can all be used to address key problems in wastewater treatment. Further, these uses of electrochemical technology have the potential to be not only used beneficially for water and waste water treatment needs but could be combined with renewable energy sources (e.g., solar energy) to transition away from fossil fuel based energy use and to enable a more diverse application space for electrochemical technology. In particular, for water and wastewater treatment needs in rural, agricultural, and remote locations or in other scenarios where non-chemical treatment options are needed, electrochemical technologies will be able to play a larger role if renewable energy sources can be used to meet the energy requirements of the technology.

Our research is currently focused on several wastewater treatment problems that might be solved through an electrochemical engineering approach. One particular focus of our research is the use of an electrochemical process to recover and recycle nitrogen and phosphorus nutrients in wastewater as a reusable fertilizer product. In this research, we focus on the formation of struvite, which is hydrated magnesium ammonium phosphate (Hug, A.

and Udert, K.M. 2013, Kékedy-Nagy, L. et al. 2019). While there has been extensive research on the chemical precipitation of struvite via the addition of a magnesium salt (e.g., magnesium chloride) and base (e.g., sodium hydroxide) for pH elevation (e.g., (Kim, D. et al. 2007, Wilsenach, J.A. et al. 2007, Ye, Z.-L. et al. 2011, Hutnik, N. et al. 2013, Zhang, T. et al. 2014, Agrawal, S. et al. 2018, Li, B. et al. 2018, Tansel, B. et al. 2018)), the use of an electrochemical system to cause struvite precipitation remains little studied (Ben Moussa, S. et al. 2006, Wang, C.-C. et al. 2010, Hug, A. and Udert, K.M. 2013, Kruk, D.J. et al. 2014, Belarbi, Z. and Trembly, J.P. 2018, Kékedy-Nagy, L. et al. 2019). In the electrochemical approach, a magnesium anode is used, and the corrosion of the magnesium metal to magnesium cation in aqueous wastewater solutions drives struvite precipitation. Key parameters in the electrochemical system include the composition of the magnesium electrode, water composition, and pH. In the present study, we focus our initial experiments on a synthetic water solution of aqueous ammonium phosphate, and we comparatively evaluate a pure magnesium electrode against an AZ31 magnesium alloy; all other parameters are held constant.

Additional interests in our research group include the use of electrochemical approaches to address the removal of key contaminants and enable a chemical-free disinfection step for remote and rural wastewater treatment needs in the agriculture and aquaculture sectors. In particular, we are focused on electrode electrocatalyst design and electrochemical cell design for the treatment of aquaculture water. Our research target is simultaneous electrooxidation of ammonia and the production of hydrogen peroxide for disinfection. We use graphite electrodes as the base electrode material (Gupta, N. and Oloman, C.W. 2006, Zöllig, H. et al. 2015, Peera, S.G. et al. 2016). The purpose of the graphite electrode as the anode is to promote the electrooxidation of ammonia to nitrogen gas (Zöllig, H. et al. 2015), while the role of the graphite electrode as the cathode is as a poor electrocatalyst for the oxygen reduction reaction to water, where dissolved oxygen will only be partially reduced at the graphite surface to hydrogen peroxide ( $H_2O_2$ ) rather than  $H_2O$  (Gupta, N. and Oloman, C.W. 2006). Ammonia oxidation at the anode can be enhanced by the presence of precious metals such as platinum and ruthenium (Donten, M. et al. 1997, Bertin, E. et al. 2012, Jiang, J. 2017); while these metals are more expensive than a graphite-only electrode, the enhancement in the electrooxidation reaction provides a potential trade-off between energy required versus electrode cost. In this paper and associated talk, we discuss our ongoing work and recent results on electrochemical approach, electrochemistry experiments, and struvite precipitate characterization for the recovery and recycling of ammonium and phosphate nutrients as the slow-release fertilizer compound struvite (magnesium ammonium phosphate hexahydrate) from simulated wastewater. We also discuss initial experimental results for the simultaneous ammonia oxidation and disinfection of aquaculture water by conducting experiments in simulated sea water.

## 2. Experimental Approach

### 2.1 Electrochemical Struvite Precipitation

Experiments were performed with aqueous solutions containing 0.077 M (7.53 g  $L^{-1}$ ) dihydrogen ammonium phosphate ( $NH_4H_2PO_4$ ) from Sigma-Aldrich. The test solutions were prepared by using Milli-Q water (18.2 MΩ, Millipore, Bedford, MA, USA). The magnesium foil (99.9% pure), AZ31 magnesium alloy foil (Al 3 wt%, Zn 1 wt%, balance Mg) and stainless-steel (316SS) plates of dimension 10 x 10 cm and 2 mm thick were purchased from Goodfellow Corporation.

Electrolysis experiments were conducted in a single-compartment reactor filled with 0.85 L of test solution and continuously stirred at approximately 240 rpm. The schematic illustration of the reactor setup is given in Figure 1, where the electrodes were shaped as thin plates with an active surface area of 40  $cm^2$ . Pure-Mg or an AZ31 magnesium alloy served as the anode, and 316SS served as the cathode, while the distance between the electrodes was held constant at 5 cm. The anode potential was controlled with a potentiostat ((Bio-Logic, USA) and was measured against a double-junction Ag/AgCl (3M NaCl, BASi) reference electrode, where the reference electrode chamber was set close to the anode surface. Single batch experiments were conducted with each test lasting 6 hours at a fixed anode potential of -0.8 V (Kékedy-Nagy, L. et al. 2019). After each batch experiment, the precipitate from the anode and the cathode were collected and stored separately. The precipitate from the test solution was recovered by vacuum filtration, where the filter holder was fitted with PTFE un-laminated membrane filters (0.45-micron, 47 mm from Sterlitech). Precipitates were characterized by powder x-ray diffraction (XRD) and scanning electrochemical microscopy (SEM).

### 2.2 Electrooxidation of Ammonia with Simultaneous Disinfection

Experiments were performed in a simulated sea water aqueous solution (Kester, D.R. et al. 1967). The ultimate

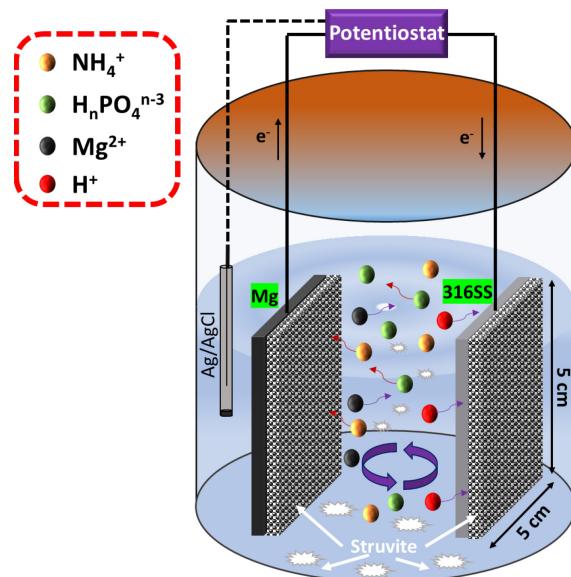
goal for this research is to perform experiments in a flow cell reactor design (Figure 5), but all data presented in this paper were collected in a batch reactor setup, split-cell design. In the split-cell design (Figure 10a), the anode is separate from the cathode by a solid porous separator. The split-cell design allows the reactions that occur at the anode and the cathode to proceed without interference from each other. The reference electrode is placed close to the working electrode (in this study, the anode) to minimize electrical resistance contributions to measured current. All electrochemistry experiments were conducted with a silver-silver chloride (Ag/AgCl) reference electrode (3 M NaCl).

For ammonia oxidation experiments, cyclic voltammetry (CV) experiments were performed between a voltage window of -1.5 and 1.5 V vs. Ag/AgCl for graphite-only electrodes and between a voltage window of 0.05 and 1.2 V vs. Ag/AgCl for graphite-supported PtRu film electrodes. PtRu films were synthesized via an electrodeposition approach with the following conditions: 10 mM  $\text{H}_2\text{PtCl}_6$  and 20 mM  $\text{RuCl}_3$  in 50 mM aqueous  $\text{H}_2\text{SO}_4$ . The deposition potential (no internal resistance correction) was -0.6 V vs. Ag/AgCl, and the deposition times were 1 min, 3 min, and 5 min. For experiments to evaluate the disinfection potential of the cathode (graphite), a commercial test kit for detection of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) concentration via color change was used. Test kit strips were dipped into the cathode compartment of the electrochemical split cell every 10 min for a 60 min experiment, and the color change of the test strip from white to specific shades of blue indicated the  $\text{H}_2\text{O}_2$  concentration in ppm (i.e., mg/L). For disinfection potential experiments, the working electrode potential was held at 1.5 V vs. Ag/AgCl for 60 min.  $\text{H}_2\text{O}_2$  is expected to form due to the partial reduction of dissolved oxygen; graphite is known as a poor oxygen reduction reaction electrocatalyst, where the full oxygen reduction reaction involves the reduction of oxygen to water.

### 3. Results

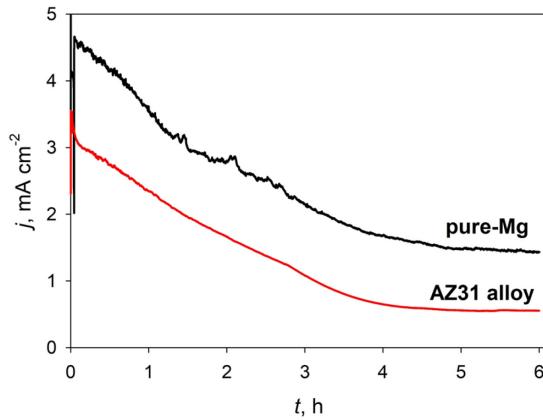
#### 3.1 Electrochemical Nutrient Recovery from Wastewater

While chemical precipitation of struvite is well-known and currently being pursued at the scale up and commercialization levels (e.g., by the company Ostara and others), electrochemical precipitation of struvite is less well-known and the details of the electrochemical precipitation process need to be studied and understood. Our goal with our current research activities is to study struvite precipitation in an electrochemical reactor setup, where struvite formation is driven by the corrosion of a magnesium-based electrode in aqueous solution containing ammonium and phosphate. Our work is focusing on understanding the fundamentals of struvite formation, particle growth and morphology, and electrochemical efficiency of the process. In Figure 1, a cartoon schematic of our batch reactor setup is shown; for the results presented below, a simple synthetic solution of ammonium phosphate was used, and the experiments were performed at the natural pH of ammonium phosphate, measured at 4.5 (Kékedy-Nagy, L. et al. 2019).



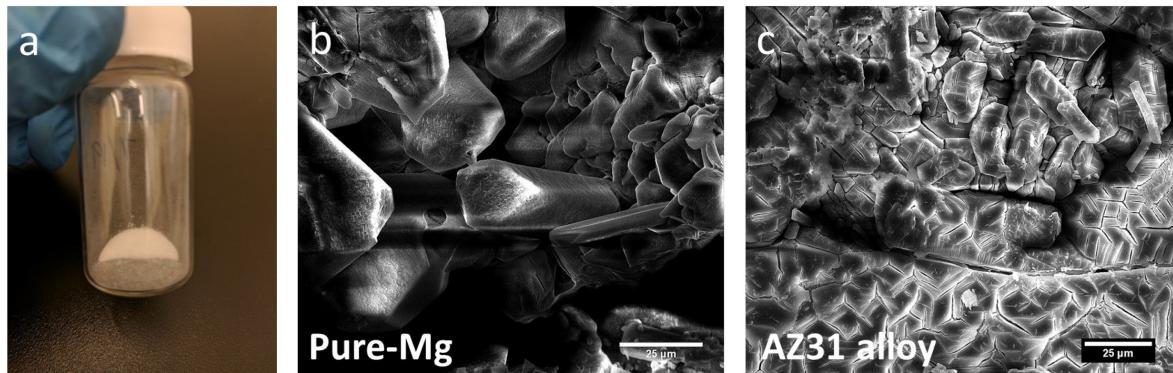
**Fig. 1: Cartoon schematic of the batch reactor setup for electrochemical precipitation of struvite from the beneficial corrosion of a magnesium anode. A Ag/AgCl reference electrode and a stainless steel cathode were used for all experiments.**

Two different electrode compositions, pure magnesium (pure-Mg) and a magnesium alloy (AZ31) were evaluated over a 6-hour experiment, where the applied voltage was held at -0.8 V and the current density ( $j$ ) was measured. Results, shown in Figure 2, demonstrate that the pure-Mg electrode results in a larger measured current density, as compared to the AZ31 alloy electrode. This result suggests that the pure-Mg anode results in a greater extent of magnesium metal corrosion and possibly a greater amount of struvite formation. Further, both anode materials result in a significant decrease in the measured current over time during the first 4 hours of the experiment; after approximately 4 hours, the measured current stabilizes and becomes relatively linear. This decline in measured current density may be the result of the formation of a passivating foulant layer on the surface of the anode, which is likely to be comprised of struvite particles.



**Fig. 2: Potentiostatic experiments, where current density ( $j$ ) is measured over time (hours), for two types of magnesium electrodes: pure magnesium and the magnesium alloy, AZ31.**

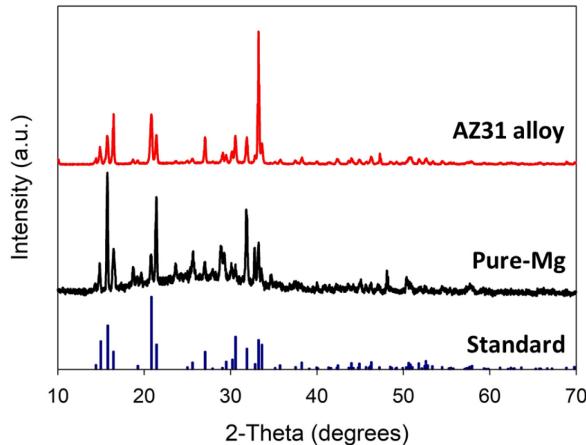
Precipitated struvite appears visually as a white particulate powder when dried (Figure 3a). Characterization results by electron microscopy, shown in Figure 3b & c, reveal large micrometer-size particulates for both anode types. SEM imaging demonstrates that the struvite particles have an elongated, rhombohedral-like structure with particle sizes ranging from 15 – 30  $\mu\text{m}$ . Notably, the particles produced by the pure-Mg anode appear more loosely packed, while the particles produced by the AZ31 alloy appear to be more densely packed.



**Fig. 3: (a) Digital image showing struvite powder. (b, c) X-ray diffraction (XRD) spectra for electrochemically-precipitated struvite from the pure magnesium anode and from the AZ31 magnesium alloy anode.**

For both anode types, the precipitates formed resulted in a similar crystal structure to a chemically-precipitated struvite reference standard, as measured by x-ray diffraction (XRD) (Figure 4). The slight differences in XRD spectra between electrochemical and chemical struvite and between the precipitates formed from the two different anodes suggest that different crystal faces dominate amongst the different samples. This result confirms the presence of struvite in the precipitate recovered from each experiment, and the lack of other diffraction peaks in the experimental spectra suggest that struvite was the primary, if not the only, crystalline structure obtained. This result is expected based on the simple synthetic solution used in experiments, which contained only ammonium and phosphate, as well as based on the relatively low starting pH (4.5). The low starting pH precludes the formation of precipitates that might be expected at higher pH values (e.g., magnesium hydroxide or magnesium phosphate)

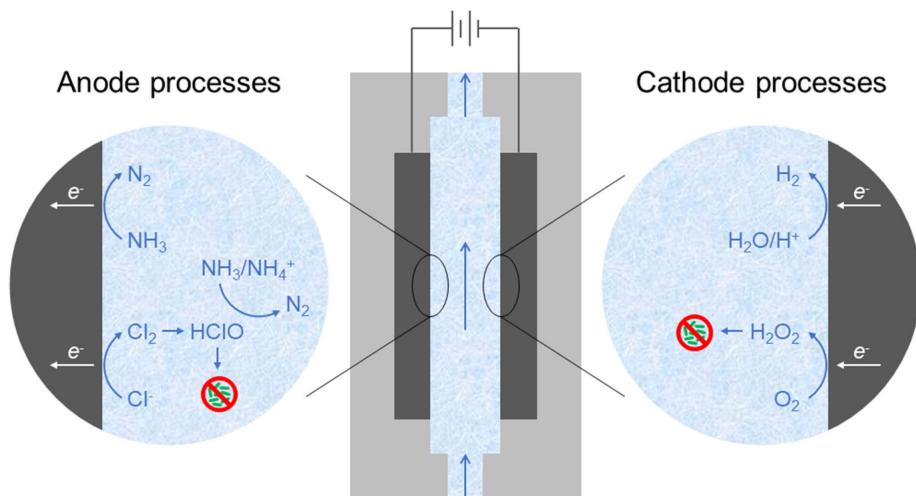
(Hug, A. and Udert, K.M. 2013).



**Fig. 4: X-ray diffraction (XRD) results for the pure magnesium and AZ31 magnesium alloy used as the anode to drive electrochemical struvite precipitation, compared to a standard reference. Both anodes resulted in a precipitate with the crystal structure of struvite.**

### 3.2 Electrochemical Treatment of Aquaculture Water: Ammonia Oxidation

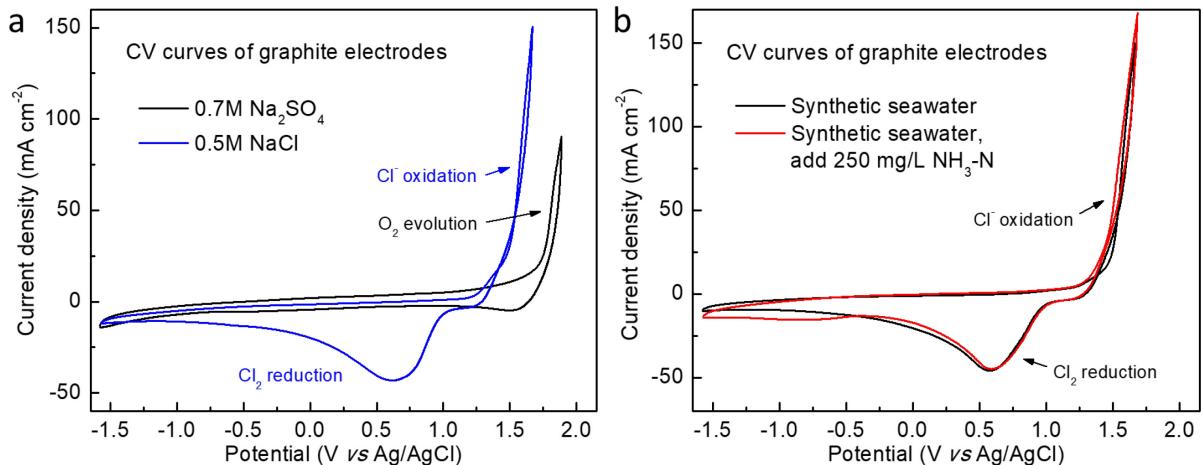
An electrochemical approach can also be used to address specific contaminant and treatment target needs, such as chemical-free disinfection and the oxidative removal of aqueous ammonia. Our research for this application is focused on aquaculture systems, where aqueous ammonia builds up as a result of the natural biological processes of the fish living in an aquaculture system. Disinfection of aquaculture water is desirable and required due to the presence and build up of bacteria and other potential pathogens (e.g., viruses), and a non-chemical (i.e., chlorine-free) approach better enables direct recycling of the aquaculture water back to the system. Our research focuses on the eventual development of a flow-based electrochemical reactor, where ammonia oxidation occurs at the anode and the production of  $\text{H}_2\text{O}_2$  occurs at the cathode, enabling disinfection (Figure 5). Disinfection may also occur at the anode due to chloride anion oxidation to hypochlorite.



**Fig. 5: Cartoon Schematic of electrochemical approach to aquaculture water treatment, where the anode design targets ammonia oxidation and the cathode design targets the production of hydrogen peroxide for disinfection.**

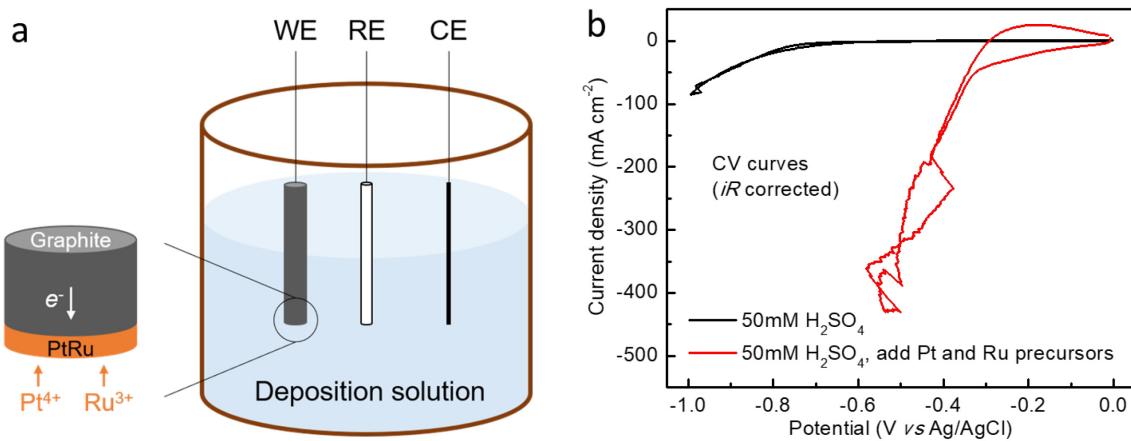
Our initial electrochemical studies were performed in a non-flow batch reactor to first conduct a fundamental evaluation of electrode performance in the presence of different electrolytes and aqueous ammonia. Our initial studies focused on an evaluation of graphite electrodes, based on prior work (Zöllig, H. et al. 2015). A set of graphite electrodes were first evaluated in a sodium sulfate electrolyte in comparison to a sodium chloride electrolyte (Figure 6a) to understand the behavior of the graphite electrode in the presence and absence of chloride. From these cyclic voltammetry (CV) results, we observe the onset of chloride oxidation at approximately 1.2 V, as

well as a significant current increase caused by the oxygen evolution reaction (OER) starting at approximately 1.5 V (blue curve, Figure 6a). In comparison, in the absence of chloride, only the OER is observed. The addition of ammonia to a simulated seawater electrolyte (Figure 6b) results in subtle changes to the CV curve produced by the graphite electrode; the majority of the current produced is likely from direct oxidation of chloride and then the OER. This result suggests that in the presence of chloride enables indirect chemical oxidation of ammonia, where the chloride is oxidized at the electrode surface to hypochlorite, and the hypochlorite then oxidizes the ammonia molecules. This ammonia oxidation reaction converts the hypochlorite back to chloride anion and likely only occurs close to the electrode surface.



**Fig. 6:** (a) Cyclic voltammetry (CV) results for a graphite electrode in two different electrolytes. The presence of chloride anion results in a shift in the onset of current production to lower voltage, indicative of chloride oxidation. (b) CV results for a graphite electrode and synthetic seawater with and without ammonia.

To evaluate the potential use of a PtRu film as an electrocatalyst layer on a graphite support, electrodeposition of PtRu was performed as illustrated in Figure 7a. The PtRu films were thin as electrodeposition was performed for short times (1 – 5 min). Electrodeposition of the film was followed by measurement of the current density produced; the result in Figure 7b (red curve) is a representative dataset illustrating the current measured during Pt/Ru deposition, as compared to a control experiment for graphite in the presence of the background sulfuric acid electrolyte with no Pt/Ru precursors added (black curve).



**Fig. 7:** (a) Cartoon schematic of electrodeposition approach for applying a thin PtRu film to a graphite electrode. (b) CV measurement during PtRu film electrodeposition (red curve), in comparison to a control (black curve).

The PtRu-graphite electrodes were then evaluated by a set of CV experiments in simulated sea water in comparison to graphite alone (Figure 8) for different aqueous ammonia concentrations. The results shown in Figure 8 for 0 and 250 mg/L NH<sub>3</sub>-N demonstrate that the presence of the PtRu film drastically reduces the required applied voltage for the onset of oxidative processes to occur. In particular, the onset of the OER can be observed at  $\sim 1.2$  V as a steep increase in current with voltage, but prior to the onset of the OER, there is a broad oxidative feature that

appears and increases in current density with increasing PtRu film deposition time. Within this feature, there is an observable peak at 0.7 V vs. Ag/AgCl.

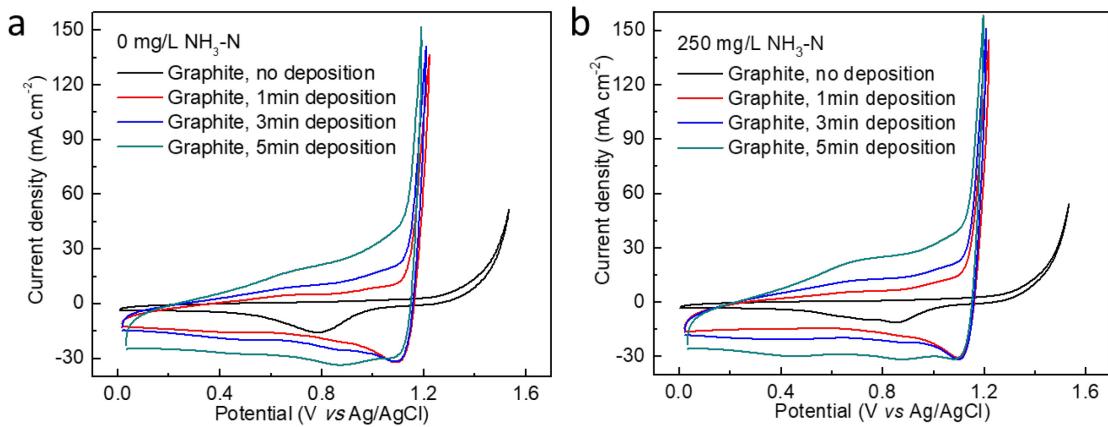


Fig. 8: Graphite and PtRu film-graphite electrodes evaluated in simulated sea water solution in the presence of (a) no aqueous ammonia and (b) 250 mg/L NH<sub>3</sub>-N added aqueous ammonia. PtRu films were electrodeposited for 1 min, 3 min, or 5 min.

When the CV results for several concentrations of aqueous ammonia are plotted together (Figure 9, for a 5 min electrodeposition time), we observe that the peak identified at 0.7 V vs. Ag/AgCl increases in current density with an increase in aqueous ammonia concentration. This result suggests that ammonia is oxidized at this voltage; further experiments are required to understand the reaction pathway and resulting oxidation products of electrooxidation of ammonia by these PtRu films versus graphite only electrodes. It is important to note that the voltage at which ammonia is oxidized when the PtRu films are used (0.7 V) is much lower than that of the graphite-only electrodes (~1.4 V), based on these CV studies. This result suggests that there will be a trade-off between the energy required to oxidize ammonia (as determined by the voltage required and the current density produced, where current multiplied by voltage equals Watts) and the cost of the electrocatalyst on the electrode. In addition, the stability of the electrode will need to be evaluated, but precious metals such as Pt and Ru are known to be relatively stable, especially at lower voltages. However, graphite can be oxidatively corroded when used at higher voltages, which would be required to achieve ammonia oxidation. These trade-offs between energy use and electrode cost and performance and stability will need to be evaluated in future work.

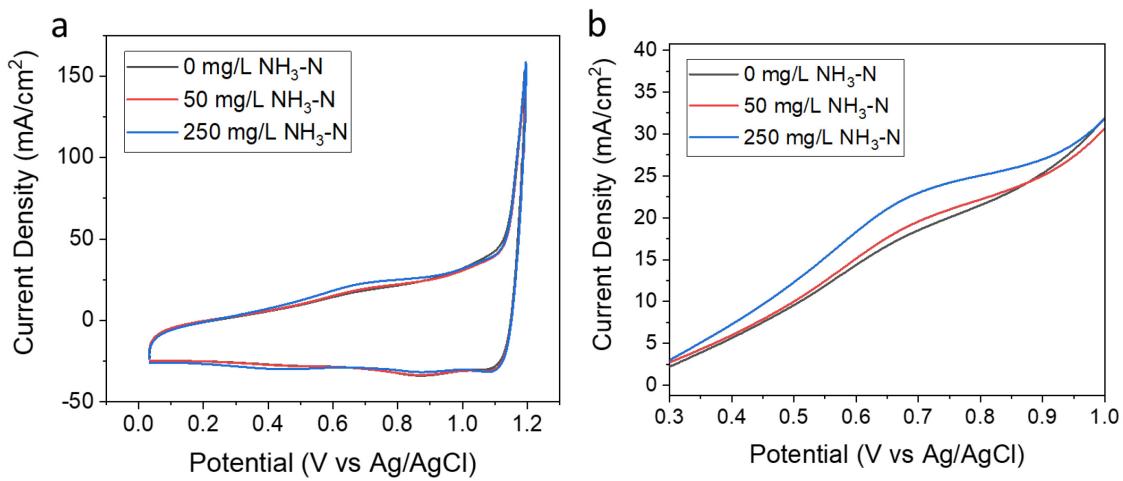
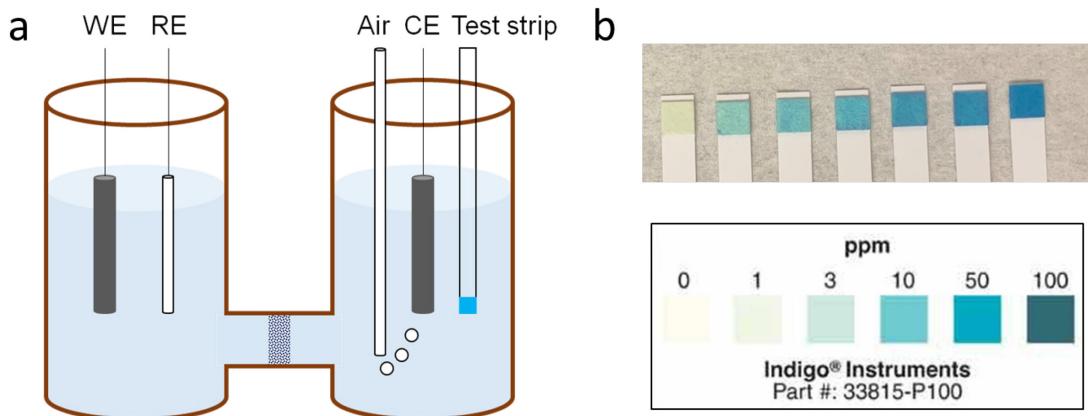


Fig. 9: (a) CV results for a PtRu-graphite electrode synthesized with an electrodeposition time of 5 min as a function of aqueous ammonia concentration. The full voltage window of the experiment, 0.05 V – 1.2 V vs. Ag/AgCl, is shown. (b) At 0.7 V vs. Ag/AgCl, an ammonia oxidation feature is identified, where the current density response increases as aqueous ammonia concentration increases. All experiments were performed in simulated sea water solution.

Finally, preliminary experiments have been performed to confirm and qualitatively quantify the production of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) at the graphite cathode in the split-cell electrochemical setup (Figure 10a). A

commercial test kit with color-change test strips was used, and results for a 60-min experiment are shown in Figure 10b (top image), compared to the test kit color-concentration correlation key (bottom image). The experimental test strips shown in Figure 10b were each dipped into the cathode (i.e., counter electrode (CE)) compartment every 10 min, and the observed color change shows the gradual increase in H<sub>2</sub>O<sub>2</sub> concentration (ppm) over time, where a concentration of ~50 ppm H<sub>2</sub>O<sub>2</sub> in solution was reached at ~40 min. We anticipate that in a flow cell, with operational parameters optimized, a steady-state H<sub>2</sub>O<sub>2</sub> concentration can be reached, and with changes in flow cell parameters (e.g., flow rate, residence time), the H<sub>2</sub>O<sub>2</sub> concentration can be controlled at the desired target concentration.



**Fig. 10:** (a) Cartoon schematic of experimental setup to evaluate the production of hydrogen peroxide at the graphite counter electrode (i.e., the cathode). (b) In the top image, results from an electrochemical experiment are shown, where a test strip was used to sample the counter electrode compartment every 10 min over 60 min. In the bottom image, the indicator comparison from the commercial hydrogen peroxide test kit is shown, with color-concentration key. WE = working (anode) electrode; RE = reference electrode; CE = counter (cathode) electrode.

#### 4. Acknowledgments

LKN and LFG acknowledge the US National Science Foundation (NSF) for financial support of this work through the INFEWS/T3 Award # 1739473. GQ and LFG acknowledge the Hawaii Farm Bureau and the Hawaii Dept. of Agriculture for financial support of this work through grant # 65587. The authors acknowledge the University of Arkansas Institute for Nanoscience and Engineering for support and access to characterization instrumentation.

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