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Project R-122 Q0

An Introduction to AESF Research Project #R-122

Electrochemical Approaches to Treatment of PFAS in Plating Wastewater

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Editor's Note: For 2021, NASF-AESF Foundation Research Board has selected a project on addressing the problem of PFAS and related chemicals in plating wastewater streams. This report introduces the project and outlines the work to be undertaken.

1. Introduction and Significance of Activity to NASF

Per- and polyfluoroalkyl substances (PFASs) are a class of diverse organic chemicals that have been used for decades in a wide variety of applications and products. These chemicals are highly surface active and have extreme thermal and chemical stability that stem from the high energy carbon-fluorine bonds (531.5 kJ/mol) and the helical conformation of their molecular structures.¹⁻⁴ As such, PFASs have been used as surface coatings, fire retardants, surfactants and lubricants for a number of applications, including metal plating, semiconductors, textile and firefighting. The widespread use of PFASs in combination with their stability has resulted in their ubiquitous presence in the environment. PFASs have been frequently detected in natural water bodies as well as in drinking water worldwide.^{5,6} The public concern over PFASs has heightened because of their global distribution,⁵ environmental persistence, bioaccumulation^{7,8} and potential toxicity.⁹⁻¹¹ The USEPA has issued a Health Advisory Level for perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS) of 70 ng/L, individual or combined.¹²

PFOS has been used for metal finishing and electroplating applications as mist suppressant, usually added in its tetraethylammonium salt form.^{13,14} PFOS has been gradually replaced by other polyfluoroalkyl substances that have similar mist suppressing ability, but are relatively easier to degrade, such as 6:2 fluorotelomer sulfonate (6:2 FTS) and 6:2 monochlorinated-polyfluorinated-ether sulfonate (F-53B). They are thus present in the plating wastewater at tens of $\mu\text{g/L}$ levels, orders of magnitude greater than the EPA health advisory levels. The toxicity of 6:2 FTS and F-53B has also been documented,¹⁵⁻¹⁷ although they have not been studied as extensively as that of PFOS. Besides, 6:2 FTS and F-53B may seem more readily degradable than PFOS, but likely they may only undergo partial degradation to chop off the nonfluorinated segments and end up as the more persistent perfluoroalkyl acid (PFAA) counterparts. As such, treatment of PFAS present in plating wastewater is challenging, whether they are PFOS or the replacement 6:2 FTS and F-53B.

The technologies applicable to remove PFASs from water can be categorized into two types: the physical approaches based on adsorption and separation and the chemical methods involving PFAS destructions.⁴ Adsorption by granular activated carbon (GAC) is widely used for the removal of PFOS and PFOA in groundwater at contaminated sites but is less effective for PFAAs of shorter carbon chains. Ion exchange resins (IXR) are effective for both long- or short-chain PFAAs, while their effect on nonionic PFASs and the influence of ions in natural waters need investigation. Our recent study¹⁸ as well as studies by other researchers¹⁹⁻²¹ have indicated that electrocoagulation (EC) can generate zinc or iron flocs *in situ* that are able to strongly adsorb certain PFASs, such as PFOA and PFOS. Reverse osmosis (RO) and nanofiltration (NF) have been shown effective in removing PFASs regardless of chain length.¹⁷ This approach generates a low volume, high concentration retentate waste which requires treatment or disposal, as these processes do not destroy PFASs.

Destruction of PFASs for treatment purpose is challenging, particularly for PFAAs. The extreme chemical stability of PFAAs renders them highly resistant to conventional treatment technologies such as advanced oxidation processes (AOPs). AOPs rely primarily on hydroxyl radicals ($\cdot\text{OH}$) to destroy organic contaminants, but the relatively slow reaction rates between PFAAs and aqueous $\cdot\text{OH}$ limit their applicability.²²⁻²⁴ Some technologies have shown limited success in degrading PFAAs in laboratory-scale studies. For example, some photolytic techniques have shown varying degrees of efficacy on high concentrations of PFAAs, including direct UV photolysis, photochemical oxidation with $\text{K}_2\text{S}_2\text{O}_8$, $\text{H}_3\text{PW}_{12}\text{O}_{40}$, KI, and humic acid (HA), and photocatalysis in



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the presence of TiO_2 and In_2O_3 .²⁵⁻³⁰ Ultrasonic irradiation and plasma oxidation have shown effectiveness to degrade PFAAs.^{31,32} Reduction by zero valent iron under sub-critical water conditions has led to possible PFOS degradation.³³ Applications of these technologies are, however, limited by their requirement of high energy input and/or special equipment.

Electrochemical oxidation (EO) has been shown promising for PFASs treatment in water. It is particularly effective to destroy PFASs involving a mechanism by direct electron transfer and indirect free radical reactions working in concert.^{34,35} EO is a favorable PFAS destruction technology also because it functions at room temperature and atmospheric pressure without added reagents.^{29,30,36-39} In addition, EO treatment is easy to manipulate and automate, and is thus suitable for distributed water treatment applications.⁴⁰ The materials to be used as anode in EO water treatment are rare, because they have to meet a few stringent requirements in stability, conductivity and electrocatalytic activities. Most previous studies on EO treatment of PFASs were conducted using boron-doped diamond (BDD) electrodes that cannot be readily fabricated at large scale, thus prohibiting its field application.^{36,38}

Our recent studies have shown that Magnéli phase titanium sub-oxides (TSO) can be used as suitable anodes to effectively degrade PFAAs in EO.⁴¹⁻⁴³ These materials, comprising a series of distinct compounds having the generic formula $\text{T}_{1-x}\text{O}_{2n-1}$ ($3 < n < 10$), have recently been explored as promising candidates for electrochemical applications because of their high conductivity, chemical inertness and facile production.⁴⁴⁻⁴⁷ TSO can serve as ideal anodes in EO water treatment also because they have large overpotentials with respect to water decomposition, providing a wide electrochemical window ($-1.4 \text{ V} \sim 2.0 \text{ V}_{\text{SCE}}$) for chemical redox reactions.⁴⁸

In particular, TSO can be readily made into porous ceramic materials that enable reactive electrochemical membrane (REM) operation, in which the anode also serves as a membrane simultaneously, which greatly enhances EO efficiency in comparison to the batch operation mode.⁴⁹ This is because the active electrochemical surface available for reaction is greatly increased as the contaminated water is filtered through the anode, and the interphase mass transfer is also improved via convection-enhanced dispersion. Recent studies indicate that TSO-based REM enables rapid PFAS degradation even by just single pass of the contaminated water.⁴¹ Our study also indicates that the conversion of chloride to chlorate and perchlorate, a concern commonly associated with EO water treatment, is significantly slower on Ti_4O_7 anode than the widely studied BDD anode.⁴³ This is because chloride does not undergo direct electron transfer on Ti_4O_7 anode, unlike BDD anode.

2. Related Work

Dr. Huang's laboratory at the University of Georgia has conducted research for years on innovative approaches to the destruction of PFASs in water and soil funded by DoD's Environmental Research Programs and U.S. EPA. In a recent study, Huang's lab has fabricated porous Ti_4O_7 anodes by high temperature sintering techniques. Such anodes were found to facilitate rapid PFOS degradation with nearly stoichiometric release of fluoride, indicating its complete mineralization. Such anodes were also tested in REM operation that is schematically indicated in Fig 1, and the results were also encouraging. The energy efficiency per order of PFOS degradation (EE/O) was compared between the batch reactor treatment and the REM operations at varying anodic potentials (Fig 2). While it is evident that the REM operation is advantageous with regard to energy efficiency, batch reactor operation may still be necessary in scenarios where REM treatment is hard to implement. Our studies have also demonstrated that the porous Ti_4O_7 anodes are highly efficient to degrade a number of PFASs other than PFOS in electrooxidation.⁴²

One alternative method to improve the interphase mass transfer of PFAAs during EO is to concentrate them prior to electrolysis. It appears that an EC process we recently developed¹⁸ may serve such a purpose well. We have found that PFAAs, such as PFOA and PFOS, can be quickly adsorbed on the surface of zinc hydroxide flocs generated by EC, mainly via hydrophobic interaction with sorption capacity (q_e) up to 5.74/9.69 mmol/gm (Zn) for PFOA/PFOS and an initial sorption rate of $1.01 \times 10^3 / 1.81 \times 10^3 \text{ mmol gm-hr}$. The EC-generated zinc hydroxide flocs have a much higher sorption capacity and faster sorption rate than other sorbents reported in previous studies or preformed zinc hydroxide. For example, the sorption capacity and rate of PFOA on GAC were reported to be 0.39 mmol/gm and $1.06 \times 10^{-2} \text{ mmol/gm-hr}$, respectively.⁵⁰ These advantages enable EC-generated zinc hydroxide flocs to effectively adsorb PFAAs from water within a short hydraulic retention time. Unlike the other sorbents, metal hydroxide flocs can be easily dissolved in controlled volumes of acid solutions, so that the adsorbed PFAAs are released back to solution and thus concentrated. This feature is important to enable the coupling between EC and EO. The EC process concentrates PFAAs in a small volume of acid solution, therefore reducing the energy consumption and

cost of EO treatment. EC involves low energy consumption and has been widely used in water treatment for decades, thus allowing for easy scaling-up.

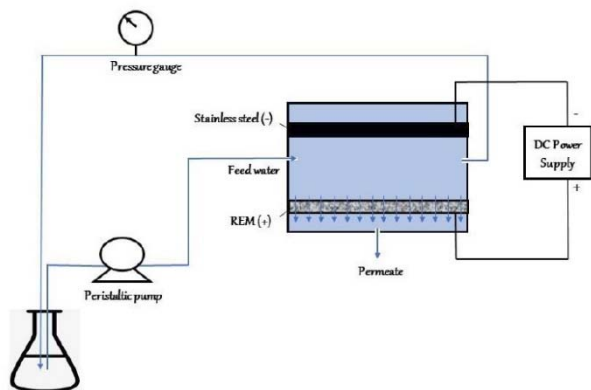


Figure 1 - A schematic diagram of REM reactor with Ti_4O_7 anode in cross-flow filtration operation.

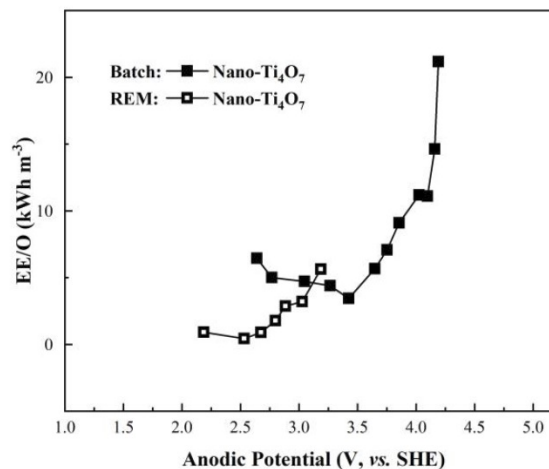


Figure 2 - EE/O in relation to the anodic potential for PFOS degradation during EO treatment in REM or batch reactor with Ti_4O_7 anode.

3. Technical approach and planned tasks

3.1 Technical approach

Plating wastewaters contain high concentrations of ions and thus lend themselves particularly amenable to electrochemical treatment, such as electrooxidation (EO) and electrocoagulation (EC). EC generates metal hydroxide flocs that adsorb PFASs and separate them from water, while EO destroys PFASs in water by direct electron transfer and hydroxyl free radical attacks in concert. They thus meet different treatment goals and are suitable for different situations. In particular, metal hydroxide flocs can be easily dissolved in controlled volumes of acid solutions, thus concentrating the PFAAs that can be subsequently destroyed by EO more efficiently. This EC-EO treatment train is particularly useful for plating wastewater treatment, because no electrolyte modification will be necessary for the entire process. The metal ions contained in the concentrate can be removed from water by depositing on the cathode during the EO process. Any remaining ions can be readily removed by precipitation with sulfide.

This research includes three research tasks that are designed to investigate EC, EO and EC-EO treatment trains, respectively, with regard to PFAS removal in plating wastewaters. These tasks are designed to probe three hypotheses specified follows:

1. EC generates amorphous metal hydroxide flocs that can effectively adsorb PFASs in plating wastewater, which, through an appropriate treatment, can release PFASs into a concentrated solution.
2. EO enabled by a Magnéli phase Ti_4O_7 anode can be used to effectively destroy PFASs in plating wastewater.
3. The electrochemical treatment train comprised of EC and EO by Ti_4O_7 anode can remove and degrade PFASs in plating wastewater more efficiently than either process operated individually.

In all tasks, the samples will be prepared by spiking water and/or synthetic plating wastewater with PFOS, 6:2 FTS and F-53B as the target chemicals, individually or mixed. The concentrations of PFASs will be quantified using a Waters ACQUITY I class ultra-performance liquid chromatography system coupled with a XEVO TQD triple quadrupole mass spectrometer (UPLC-MS/MS, Waters Corp., Milford, MA) in negative electrospray ionization mode (ESI-). The UPLC is equipped with an Acquity UPLC BEH C18 column (2.1 × 10 cm, 1.7 μm particle-size) operated at 40°C. A gradient composition of solvent A (5 mM ammonium acetate in Milli-Q water) and solvent B (5 mM ammonium acetate in methanol) will be used as mobile phase at 0.3 mL/min flow rate. Concentrations of the target PFASs will be quantified using calibration with isotope-labelled internal standards. The concentration of cations will be quantified using an ICP-MS (Perkin Elmer Elan 9000 inductively coupled plasma equipped

with a mass spectrometer). A Thermo Scientific Dionne Integriion HPIC System will be used to quantify anions in samples. Total organic carbon (TOC) will be measured using a Shimadzu TOC-Vcsh Carbon Analyzer.

3.2 Planned tasks

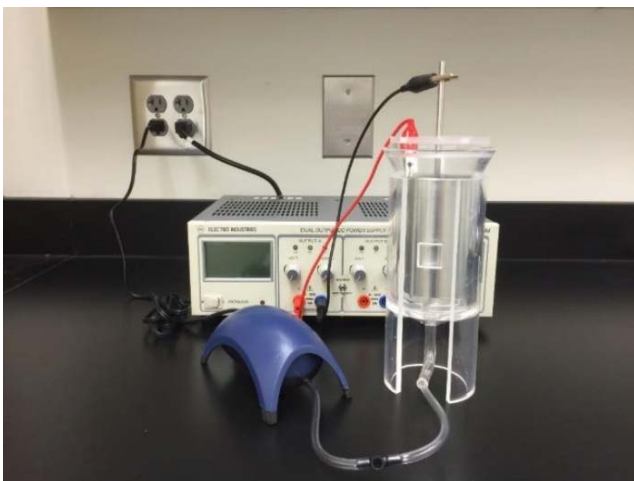


Figure 3 - Bench-scale EC reactor.

Task 1 – Removal of PFAS by EC

The EC reactor is constructed with an acrylic cylindrical EC cell (8-cm diameter and 10-cm height). A zinc or iron sheet (0.05 × 10 × 20 cm) is placed along the circumference of the cylindrical reactor to act as the sacrificial anode, while a 304 stainless steel rod of 3 mm diameter is placed at the axial center of the anode as the cathode (Fig. 3). In each treatment, 250 mL of synthetic plating wastewater will contain PFOS, 6:2 FTS and F-53B, mixed or individually. The solution will be agitated constantly throughout the experiment using an air pump through valves equipped on the bottom of the reactor. All EC experiments will be conducted in batch mode with direct current applied using a DC power source (Electro Industries Inc., Monticello, MN) at constant current density.

EC experiments will be performed with the concentration of PFASs at different levels under different current densities. Triplicate aqueous samples will be taken at the end of the EC experiment. The remaining solution will then be passed through a 0.22 μm acetate membrane (Sartorius, Germany), and the floc retained on filter will be transferred into a beaker and dissolved in 10 mL 4.5 M H₂SO₄ solution. All samples will be mixed with isotope labeled internal standard solution for subsequent UPLC-MS/MS analysis to quantify PFAS. The concentrations of F⁻ and other ions in the solution and TOC will also be quantified for selected samples. For systems tested at selected conditions, further examinations will be conducted to characterize the flocs and obtain more information regarding PFASs removal mechanisms during EC.

Task 2 – Removal of PFAS by EO

A high-temperature sintering method is used to fabricate the porous titanium suboxide (TSO) anodes to be used in batch and REM systems. Ti₄O₇ powders are produced by reducing TiO₂ powders with particle size in the nanometer range at high temperature (950°C) under a controlled H₂ atmosphere. Doping of the TSO anodes with various elements, *e.g.*, cerium, palladium and cobalt, will be attempted using hydrothermal or plating techniques. The crystalline phases of the TSO materials will be identified using an X'Pert PRO MRD X-ray diffractometer (XRD) (PANalytical, The Netherlands). The pore size and pore distribution will be obtained via mercury intrusion porosimetry by a Micromeritics Autopore IV 9500 mercury porosimeter (Norcross, GA). Anodic potential measurement, linear sweep voltammetry and cyclic voltammetry (CV) will be conducted using a CHI-660E electrochemical workstation (Austin, TX) with a leak-free Ag/AgCl reference electrode (Warner Instruments, LF-100).

For batch operation, the experiments will be carried out in 70-mL polypropylene vessels, each containing 50 mL reaction solution with PFOS, 6:2 FTS and F-53B spiked, mixed or individually. A 316 stainless steel plate of the same size and shape is used as the cathode and placed in parallel to the anode at 2.50 cm gap. The REM reactor is composed of a hollow cylindrical vessel (45.0 mm length, 60.0 mm diameter) made of acrylic, with the TSO anode installed in cross-sectional direction, and a circular 316 stainless steel plate of the same size installed in parallel at 1.20 cm gap as the cathode. A feed inlet and a retentate outlet are installed to allow sample solution flow through the space between the cathode and the anode, while another outlet is installed on one end to collect permeate filtrating through the anode. The reactor setup is illustrated in Fig 1, above.

Task 2 experiments will be performed with PFAS at different concentrations under different constant current densities. The treatment efficiency will be evaluated versus different treatment conditions as well as the anodes with different doping.

Task 3 - EC-EO Treatment Train

Task 3 is a study to combine the EC and EO operations into a sequential treatment train and evaluate its performance. The EC and EO treatment conditions will be selected based on the Task 1 and 2 results. The operation and treatment performance data



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will be collected from each process and evaluated individually and as a whole. Synthetic plating wastewater spiked with PFOS, 6:2 FTS and F-53B will be tested. The concentrations of PFAS along with major ions and TOC will be monitored along the treatment steps. It is expected that the metal ions dissolved by acid from the flocs at the EC step will be removed in the EO step by cathodic deposition. It will be monitored, and, if any remains, will be removed by precipitation with sulfide as a polishing treatment after the EO step.

The energy consumption of the entire treatment train will be analyzed in terms of energy efficiency per order of degradation (EE/O) in relation to the operation conditions. The EE/O will be assessed by comparing the EC-EO treatment train with EC or EO individual treatment scenarios. The optimized conditions will be determined based on considerations of performance-conditions relationships and the energy consumption to achieve the best cost-effectiveness.

4. Relationship to NASF Goals

PFAS is a special group of contaminants that have drawn increasing public concern and regulatory scrutiny, as a result of their extreme environmental persistence and potential toxicity. PFAS contaminants are present in metal finishing and electroplating wastewaters, and thus an effective treatment strategy is in urgent need to meet regulatory requirements expected to be tightened. Due to their special molecular configurations, destruction of PFAS for treatment purposes is highly challenging. This project, building on our recent promising studies, focuses on examining the use of EC and EO technologies in removing representative PFASs from plating wastewaters. The results will provide versatile tools to deal with PFAS-containing plating wastewaters. EC, EO or EC-EO treatment trains can be used in different scenarios to meet different treatment goals. This will greatly benefit NASF and the metal finishing and plating industry in general.

In addition, the knowledge learned in this study will advance the understanding of electrochemical processes in general, in particular, related to the conditions relevant to metal finishing and electroplating, *i.e.*, high ionic and metal contents. The different Ti₄O₇ anode materials that are modified with different doping techniques, if proven useful for wastewater treatment, will potentially provide additional business opportunities to the plating industry. In addition to the NASF Quarterly Reports, we plan to disseminate our research results by presentation at NASF/AESF Foundation SUR/FIN conferences and will publish at least one peer-reviewed journal article with the NASF acknowledged.

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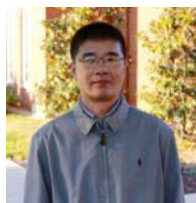


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About the author



Qingguo (Jack) Huang is Professor in the Department of Crop and Soil Sciences, University of Georgia, Griffin Campus. He holds a B.S. in Environmental Science (1990) and a Ph.D. in Chemistry (1995) from Nanjing University, China as well as a Ph.D. in Environmental Engineering from the University of Michigan, Ann Arbor, Michigan. Dr. Huang's research interest focuses on catalysis involved in the environmental transformation of organic pollutants, and development of catalysis-based technology for pollution control and environmental remediation and management. His laboratory has been actively involved in several cutting-edge research topics:

- Enzyme-based technology for water/wastewater treatment and soil remediation
- Electrochemical and reactive electrochemical membrane processes in wastewater treatment
- Catalysis in biofuel production and agro-ecosystem management
- Environmental fate and destructive treatment methods of PFASs
- Environmental application and implication of nanomaterials

He has published over 150 peer-reviewed journal articles, five book chapters and four patents and three patents pending. He has taught three courses at the University Georgia: Introduction to Water Quality, Environmental Measurement, and Advanced Instrumental Analysis in Environmental Studies.