



# **Low Energy Chemical Treatment for Graywater Reuse: Mitigating Disinfection Byproducts and Recovering Cathodically Generated Hydrogen for Energy**

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**ESTCP PROJECT ER-201637  
Final Report**

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## LIST OF ACRONYMS AND ABBREVIATIONS

| Acronym                         | Full text                                                                               |
|---------------------------------|-----------------------------------------------------------------------------------------|
| AFIOH                           | Air Force Institute for Operational Health                                              |
| ANSI                            | American National Standards Institute                                                   |
| BDCM                            | Bromodichloromethane                                                                    |
| BDD                             | Boron-Doped diamond                                                                     |
| BOD                             | Biological Oxygen Demand                                                                |
| CDBM                            | Chlorodibromomethane                                                                    |
| COD                             | Chemical Oxidation Demand                                                               |
| DBP                             | Disinfection by Products                                                                |
| DBPR                            | Disinfection by Products Rule                                                           |
| DC                              | Direct Current                                                                          |
| DoD                             | Department of Defense                                                                   |
| DSA                             | Dimensionally Stable Anodes                                                             |
| EC Cell                         | Electrochemical Cell                                                                    |
| EPA                             | Environmental Protection Agency                                                         |
| ESTCP                           | Environmental Security Technology Certification Program                                 |
| GC-TCD                          | Gas Chromatography –Thermal Conductivity Detector                                       |
| H <sub>2</sub> O <sub>2</sub>   | Hydrogen Peroxide                                                                       |
| HAA                             | Haloacetic Acids                                                                        |
| ID                              | Internal Diameter                                                                       |
| IrO <sub>2</sub>                | Iridium Oxide                                                                           |
| MMO                             | Mixed Metal Oxide                                                                       |
| Na <sub>2</sub> SO <sub>4</sub> | Sodium Sulfate                                                                          |
| NESDI BAA                       | Naval Environmental Sustainability Development to Integration—Broad Agency Announcement |
| NSF                             | National Science Foundation                                                             |
| NTU                             | Nephelometric Turbidity Unit                                                            |
| O <sub>2</sub> <sup>-</sup>     | Superoxide Radicals                                                                     |
| O <sub>3</sub>                  | Ozone                                                                                   |
| OH                              | Hydroxyl Radicals                                                                       |
| POM                             | Presidio of Monterey                                                                    |
| RO                              | Reverse Osmosis                                                                         |
| ROS                             | Reactive Oxygen Species                                                                 |
| RuO <sub>2</sub>                | Ruthenium Oxide                                                                         |
| SnO <sub>2</sub>                | Tin dioxide                                                                             |
| SS                              | Stainless Steel                                                                         |
| TCM                             | Trichloromethane                                                                        |
| TDS                             | Total Dissolved Solids                                                                  |
| THM                             | Trihalomethane                                                                          |
| TOC                             | Total Organic Carbon                                                                    |
| TSS                             | Total Suspended Solids                                                                  |
| USACHPPM                        | United States Army Center for Health Promotion and Preventive Medicine                  |
| US EPA                          | US Environmental Protection Agency                                                      |

## **1.0 INTRODUCTION**

### **1.1 BACKGROUND**

Much attention recently has been focused on the issue of water and wastewater at fixed DoD installations, forward operating bases (FOBs) and other remote DoD installations. As a result, water reuse has received increased attention. However, current water reuse systems are not optimized for energy efficiency when coupled to stringent regulations on treatment effectiveness and byproduct formation. The DoD has a prioritized need to develop and implement wastewater and water reuse treatment systems that can be operated in an energy neutral configuration and be readily deployed. Showers and laundry comprise the bulk (approximately 78%) of potable water demand at many military installations (Noblis, 2010). Sustainable reuse of the graywater resulting from these operations would substantially reduce the quantity of new water that is consumed at military installations. Thus, a technically and economically viable solution for the treatment and reuse of graywater is of great interest.

Graywater emanating from showers can contain a wide variety of contaminants, ranging from soaps, hair, dirt, pharmaceuticals, detergents, and pathogens (USACHPPM, 2008). Pathogenic bacteria emanating from fecal contamination has been identified as the primary health risk from graywater (Environment Agency, 2008). DoD guidelines state that graywater must be disinfected prior to non-potable industrial uses (e.g. flush toilets, water gardens and sometimes washing machines), while Army proposed criteria for recycled graywater for showering is based on potable water consumption standards (US Army, Navy and Air Force, 2010; USACHPPM, 2008).

### **1.2 OBJECTIVE OF THE DEMONSTRATION**

The goal of this project is to demonstrate the sustainable use of stable electrocatalytic anode materials to treat graywater for reuse in showers or laundry. Specific objectives include: (i) verification of effective destruction of chemical oxidation demand (COD), (ii) assessment of disinfection by products (DBP) formation relative to disinfection by products rule (DBPR2), and (iii) assessment of net energy requirements with respect to overall sustainability and costs. As part of objective (iii), cathodically generated hydrogen was measured to evaluate its potential for offsetting the energy requirements from the cell. Successful demonstration of these objectives was to inform the design of a pilot-scale system providing the DoD with a technically and economically feasible approach for graywater reuse.

Low BOD levels (<100 mg/L) in graywater, and other compounds such as pharmaceuticals and personal care products are not amenable to rapid biotic degradation (Leal et al., 2010; Elmitwalli et al., 2007). Current graywater treatment and reuse approaches utilize membrane filtration (microfiltration or ultrafiltration), coupled with reverse osmosis (RO) and chlorine disinfection. While the membrane/RO systems provide reliable potable water, they require expensive membranes, substantial energy input, chemical disinfection, and a concentrated contaminant reject stream that requires further treatment. (USACE, 2009). Furthermore, chlorine disinfection

can result in exceedences of the Stage 2 Disinfectants and Disinfection Byproducts Rule (DBPR2) due to formation of disinfection byproducts (DBPs) that include trihalomethanes and haloacetic acids. Recent studies have shown that inhalation risks associate with these compounds during showering are significant (Uddameri and Venkataraman, 2013), thus mitigating the generation of DBPs during graywater treatment for shower reuse is necessary. An energy efficient system for combined COD removal and disinfection of graywater that was not generate DBPs in excess of regulatory levels would address the challenges described above.

### 1.3 REGULATORY DRIVERS

The DoD recommends that potable water “be used for nearly all water-requiring activities if it is available” (US Army, Navy, and Air Force, 2010). Potable water is explicitly recommended for showering due to potential exposure via cuts, ingestion, or inhalation of aerosols. Consistent with this, the water quality criteria for recycled graywater for shower use is based on the US Army’s potable water consumption standard (USACHPPM, 2008). Recycled graywater criteria are listed in Table 1.1 below. Our proposed treatment approach for graywater reuse is therefore based on attainment of these criteria.

**Table 1.1. Water Quality Criteria for Recycled Graywater (USACHPPM, 2008)**

| Property   | Parameter           | Proposed Criteria          |
|------------|---------------------|----------------------------|
| Physical   | Color               | 50 units                   |
|            | Odor                | 3 units                    |
|            | pH                  | 5--9                       |
|            | TDS                 | 2000 mg/L                  |
|            | Turbidity           | 1 NTU                      |
|            | Chlorine (Residual) | 2--5 mg/L after 30 minutes |
| Chemical   | Arsenic             | 0.3 mg/L                   |
|            | Cyanide             | 6 mg/L                     |
|            | Lindane             | 0.6 mg/L                   |
| Biological | BOD                 | 10 mg/L                    |
|            | Coliform            | 0                          |

United States Army Public Health Command [http://phc.amedd.army.mil/PHCResourceLibrary/Non-Potable Water Reuse 2011.pdf](http://phc.amedd.army.mil/PHCResourceLibrary/Non-PotableWaterReuse2011.pdf)

An alternate graywater treatment goal is attainment of disinfection and secondary treatment standards for graywater reuse in laundry, dust suppression, or other non-potable water activities. The criteria for such uses are BOD [10 mg/L], TSS [5 mg/L], turbidity [2 NTU], non-detect total and fecal coliforms, and a chlorine residual  $\geq 1$  mg/L (USACHPPM, 2008). The US Air Force has adopted a similar graywater reuse standard for dust control (AFIOH, 2004).

## 2.0 TECHNOLOGY

### 2.1 TECHNOLOGY DESCRIPTION

Electrochemical cells (ECs) have been used to treat a wide range of organic and inorganic contaminants. The high level of treatment and relative low energy input highlight the promising nature of electrochemical treatment. The treatment mechanisms for contaminants in water include direct and indirect oxidation/reduction as well as electrocoagulation and precipitation of suspended solids. Electrolysis of water can produce several reactive oxygen species (ROS) by oxidation of water at the anode, such as hydroxyl radicals ( $\bullet\text{OH}$ ), ozone ( $\text{O}_3$ ), superoxide radicals ( $\bullet\text{O}_2^-$ ), and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Chlorine-based oxidants (e.g., hypochlorite) also can form if chloride is present in the water. These processes occur simultaneously within the EC cell, which result in the disinfection of water, destruction of contaminants, and reduction of BOD/COD and suspended solids (Jeong et al., 2007; Li et al., 2002, 2004).

The key for efficient electrolytic treatment is choice of anode material. Dimensionally stable anodes (DSA), which include the sub-category of mixed metal oxide (MMO) anodes, have high corrosion resistance and physical/chemical stability under high positive potentials. These electrodes are thermally prepared with a titanium substrate covered by metallic oxides. Corrosion resistant and chemically stable MMO anodes are frequently employed for wastewater applications. Widely used MMO anodes include titanium substrate covered by metallic oxides such as ruthenium oxide ( $\text{RuO}_2$ ) and iridium oxide ( $\text{IrO}_2$ ); these anode materials are used for COD removal and disinfection of wastewaters.

### 2.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

#### 2.2.1 Advantages

The primary advantages of performing the proposed long-term assessment approach are as follows:

The anodic treatment provides both COD removal and disinfection, and is expected to remove surfactants (Panizza et al., 2005). Unlike biological systems, flow interruptions, shock loading, or introduction of microbially inhibitory or toxic agents (e.g., anionic surfactants), was not be problematic for system operations. Electrolytic system also do not suffer from the slow removal of COD fractions that are problematic with biological approaches to graywater treatment (Ghunmi et al., 2011). Additionally, there was no waste sludge associated with electrolytic treatment as with biological treatment.

Electrochemical treatment provides a significant cost benefit over competing technologies. Schmalz et al. (2009) measured electrochemical energy consumption for disinfection and COD removal from wastewater with approximately 160 mg/L chloride, a conductivity of 1000  $\mu\text{S}/\text{cm}$ , and a pH of 7.9. The measured energy consumption was approximately 0.3 kWh/m<sup>3</sup> for a 4-log pathogen reduction and a 30 mg/L reduction in COD (Schmalz et al., 2009); decreasing the wastewater pH to below 7 reduced this energy requirement by a factor of 2. This energy demand compares very well to alternate technologies. For example, *the electrochemical energy demand (pH of 7.9) is approximately 10-times less compared to equivalent treatment using RO (Monteith*

*et al.*, 2007), and slightly less compared to UV (Blume and Neis, 2004). Costs associated with biological treatment of graywater followed by disinfection range from 0.6 to 1.2 kWh/m<sup>3</sup> (Lazarova et al., 2012), and DBP formation due to chlorine disinfection can be problematic. Other studies show that electrochemical energy consumption is similar to a coupled UV/H<sub>2</sub>O<sub>2</sub> system for removal of low COD levels (pH=7.9) (Ochiai et al., 2010; Yonar et al., 2006); Electrochemical treatment has the advantage over these systems in that oxidant addition is not required and does not have the need for lamp maintenance. In addition, hydrogen recovery would further reduce energy requirements.

### **2.2.2 Limitations**

As with all technologies and assessment approaches, there are also limitations with the proposed approach:

1. Insufficient removal of COD by filtrations prior to electrical treatment.
2. Failure of the electrodes to have a sufficient longevity during graywater treatment with scaling is likely to be the greatest cause of electrode deactivation
3. Generation of unwanted treatment disinfection byproducts (e.g., free chlorine, THMs, HAAs)
4. Potential perchlorate generation depending on electrode type and cell configuration (divided vs undivided electrochemical cells)

### 3.0 PERFORMANCE OBJECTIVES

The goal of this project was to demonstrate the sustainable use of stable electrocatalytic electrode materials to treat shower-based graywater for potable reuse. Specific objectives include: (i) verification of effective destruction of BOD/COD and pathogens in graywater under both normal and shock loading conditions to meet DoD requirements for reuse; (ii) assessment of DBP formation relative to DBPR2, (iii) demonstration of treatment longevity; and (iv) assessment of net energy requirements with respect to overall sustainability and costs. As part of objective (iv), cathodically generated hydrogen was measured to assess its potential for use in a hydrogen fuel cell to limit overall energy demand of the treatment system. Successful demonstration of these objectives will provide the DoD with a technically and economically feasible approach for graywater reuse. Performance objectives are summarized in Table 3.1.

**Table 3.1 Performance Objectives**

| <b>Performance Objective</b>                                     | <b>Data Requirements</b>                                                                                                    | <b>Success Criteria</b>                                                                                                                                                       |
|------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Quantitative Performance Objectives</b>                       |                                                                                                                             |                                                                                                                                                                               |
| Effective treatment of BOD, COD and total coliforms in graywater | Based on wastewater monitoring data collected during the 6-month operating period (especially BOD, COD and total coliforms) | Attain Water Quality Criteria for Recycled Graywater ( <b>see Table 1.1</b> ) (USACHPPM, 2008)                                                                                |
| No issues with disinfection byproducts                           | Water monitoring for trihalomethanes, haloacetic acids, and perchlorate                                                     | Demonstrate that the system can operate without exceedences of the Stage 2 Disinfectants and Disinfection Byproducts Rule (DBPR2), and perchlorate below CA regulatory levels |
| Energy efficiency                                                | Voltage, current, and treatment time, along with determination of net energy requirements (pumping, hydrogen recovery)      | <20 kw-h/m <sup>3</sup> .                                                                                                                                                     |
| <b>Qualitative Performance Objectives</b>                        |                                                                                                                             |                                                                                                                                                                               |
| System longevity and O&M requirements                            | Anode longevity, efforts to control scaling                                                                                 | O&M costs that are less than 80% of current approaches                                                                                                                        |
| Scale-up requirements for commercial applications                | Effectiveness of electrode cleaning, COD analyzers, and removal of active chlorine                                          | Mitigation of cathodic fouling, ability to effectively adjust treatment times based on COD analyzer feedback, and mitigation of active chlorine during anodic treatment       |

### **3.1 DETERMINE TREATMENT EFFECTIVENESS**

Treatment effectiveness measures how the treatment system improves the water quality. Water quality improvement is a fundamental requirement with regards to protecting the environment and enabling reuse activities.

#### ***3.1.1 Effective treatment of BOD, COD and total coliforms in graywater.***

Influent raw water quality measurements characterized the initial load of contaminants prior to treatment. Influent contaminant levels are used to assess treatment performance and determine how the demonstration results relate to other design situations. Influent raw shower water was sampled periodically from the equilibration tank upstream of the treatment system. Sampling intervals were chosen to capture potential variability in raw water quality. Key influent parameters included Chemical Oxygen Demand (COD), Total Coliform, 5-day Biochemical Oxygen, Demand (BOD-5).

#### ***3.1.2 Success Criteria***

The criteria for success were to attain water quality criteria to meet recycled graywater (Table 1.1). Published studies (Jefferson et al., 2004; Ramona et al., 2004) have shown that the COD:BOD ratio in shower-based graywater is approximately 2.5:1.

Effluent criteria included the following for average values, with 95% compliance of individual samples.

- BOD < 10 mg/L
- COD < 10 mg/L
- Total Coliform < 1 (# per 100 mL)

### **3.2 CONTROL OF DISINFECTION BYPRODUCTS**

Disinfection byproduct (DBP) formation relative to the DBPR2 and state regulations was evaluated. Specifically, formation and persistence of trihalomethanes, haloacetic acids, and perchlorate throughout the treatment processes was assessed as part of this demonstration. Residual chlorine levels also were assessed.

#### ***3.2.1 Data Requirements for DBPs.***

Treated water analyzed for trihalomethanes (chloroform, bromodichloromethane, dibromochloromethane, and bromoform), haloacetic acids (monochloroacetic, dichloroacetic, trichloroacetic, bromoacetic, and dibromoacetic), and perchlorate.

### **3.2.2 Success Criteria for DBPs.**

THM and HAA levels after treatment was compared to the DBPR2, which is a total of 80 µg/L for the THMs and 60 µg/L for the HAAs. Perchlorate levels were compared to the California MCL of 6 µg/L.

- HAA5 <60 ug/L
- TTHM < 80 ug/L
- Perchlorate <5 ug/L

## **3.3 ENERGY EFFICIENCY**

The overall energy efficiency of the graywater treatment was assessed as part of the demonstration.

### **3.3.1 Data Requirements for determining energy efficiency.**

The primary energy demand is the electrochemical cell, where the applied current, voltage and treatment flowrate will be used to determine the energy demand in W-h/L. The energy demand associated with other system components (pumps, pre-filters) also was estimated based on their energy consumption. Finally, the rate of cathodic hydrogen generation to be determined, and the rate of hydrogen production was used to calculate the potential energy recovery if a hydrogen fuel cell were installed.

- Total Energy usage <20 kw-h/m<sup>3</sup>

## **3.4 SYSTEM LONGEVITY AND O&M REQUIREMENTS**

The extent to which the electrodes remain sufficiently active with respect to COD/BOD removal, and the degree to which maintenance of the electrochemical cell and filters are needed, was be considered in the overall assessment of this technology.

### **3.4.1 Data requirements**

The COD/BOD removal rates, as well as the energy demand for COD removal, was be evaluated over the duration of the demonstration. Labor hours and any other materials cost associated with system maintenance also was be recorded.

### **3.4.2 Success criteria**

The success criteria are qualitative, but they are based on the level-of-effort and compared to alternate graywater treatment approaches. The goal is to be less than 80% of the O&M costs with current treatment approaches.

### **3.4.3 *Logic and Control Data requirements***

The ability of the currently designed logic/control process, automated electrode cleaning, and removal of disinfection by-products (including active chlorine during anodic treatment) will be carefully monitored during the 6-month duration of this demonstration. Potential issues related to operation at an increased flow rate will be carefully considered.

### **3.4.2 *Success criteria***

The success criteria are qualitative, but they are based on the effectiveness of the various control and unit operations of the overall system. NOTE: The scale-up of the electrochemical cell itself is not a key consideration of this demonstration, as commercially available cells capable of much larger treatment capacity (i.e., much greater electrode surface area) are readily available for purchase.

## **3.5 SCALE-UP REQUIREMENTS FOR COMMERCIAL APPLICATIONS**

The scale of the electrochemical treatment system is approximately 0.1 gpm. While this scale is sufficient for assessing many of the critical aspects of this technology, validation of this technology at a larger scale may be required to fully assess commercial application potential. A pilot scale electrochemical disinfection system was constructed under the Navy Environmental Sustainability Development to Integration (NESDI) Program. Results for disinfection energy required were between 0.05 to 0.2 W-h/L with no DBPs created, Schaefer et. al. 2017.



**Figure 3.1 Photo of Electrochemical Disinfection pilot system under NESDI 487.**

## 4.0 SITE DESCRIPTION

The graywater pilot system was installed outside building 627B at the Presidio of Monterey (POM) in Monterey, CA. The POM is located within the city of Monterey, California, about 117 miles south of San Francisco, on the Pacific coast. Building 627B is a barracks with more than 80 enlisted US Air Force personnel. The three-story building contains two communal, single-sex bathrooms on each floor. The pilot system was used to treat shower discharge from the female bathroom on third floor. A bank of three showers drained into a discharge manifold that penetrates the floor and runs along the ceiling of the second-floor bathroom. A site schematic that illustrates the intent of installation (Figure 4.1) in building 627B (Figure 4.2).

The pilot system piping was tied into the shower discharge manifold (Figure 4.3). The new piping was penetrated the second-floor window bank through the exhaust housing and run along the outside of the building to the pilot system located on the grass, directly outside the bathroom window. The pilot system was installed outside in an ISO or intermodal container on slope graded with 4 x 4 lumber. An electrical service was be extended from the building to the metal container and terminated in the pilot panel. Final finished water effluent was routed from the treatment system to a sewer drain located in the grass just behind the ISO container.

Building 627B - Female Wing

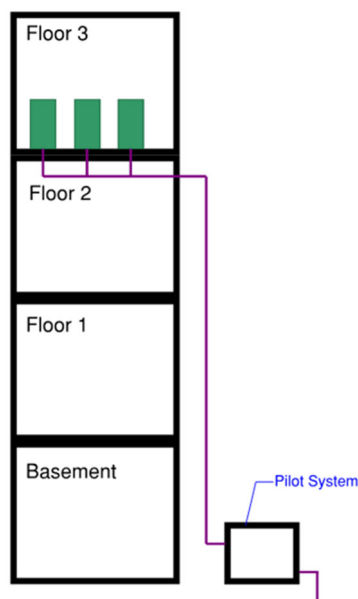


Figure 4.1. Site Schematic



**Figure 4.2. Building 627B Arrow points to showers location.**



**Figure 4.3. 3rd Floor Shower Drain and Drain Manifold. (Penetrated through floor to 2<sup>nd</sup> floor ceiling)**

## 5.0 TEST DESIGN

The following subsections provide a detailed description of the system design and testing conducted to address the performance objectives described in Section 3.0.

### 5.1 CONCEPTUAL EXPERIMENTAL DESIGN

This project focused on applying electrochemical treatment for graywater (derived from showers) reuse. Key activities for this demonstration included:

- Construction of the skid-mounted system, and site installation
- System start-up
- Bi-weekly to monthly monitoring of the graywater (raw, filtered, anodically treated, cathodically treated, and final discharge) to assess COD/BOD removal, disinfection, and DBP formation.
- Assessing energy usage, including the potential for energy recovery via cathodic hydrogen generation

The demonstration electrochemical treatment rate was approximately 0.1 gpm. The low treatment rate is due to the limited supply of graywater, and the intent to minimize electrode costs. While it is recognized that this pilot scale flow rate is much lower than typically employed in ESTCP demonstrations, the critical aspects of this technology needed for scale-up should be assessed in this demonstration and not adversely impacted by the low flow rate. These include:

1. System programming and controls to maintain effective treatment, including COD monitoring with feedback so as to not “overtreat” low COD graywater, and to effectively handle shock loading.
2. Automated electrode cleaning (including polarity reversal and acid washing) to maintain longevity over the 6-month operating period.
3. Demonstrated long-term COD removal coupled with mitigation of disinfection by-products.

These aspects of treatment assessment are the most critical and are largely independent of scale. For the commercially available electrochemical cell employed in this demonstration, increased capacity to treat a greater flow rate of graywater would simply require the purchase of larger electrochemical cells with increased electrode surface area; such cells are readily available. Because a lower flow system was used the cost per gallon treatment may be higher than a larger scale system.

### 5.2 BASELINE CHARACTERIZATION ACTIVITIES

Bench-scale testing was performed to determine the optimal electrochemical (EC) configuration and operating strategy. The details of this testing were provided to ESTCP in the Treatability Study Report and are provide here. Key results from this testing showed that:

- A divided EC cell configuration, using sequential anodic-cathodic treatment, was advantageous for COD removal, mitigating DBPs, and limiting overall energy demand.
- DBPs were reductively treated at the cathode.
- The low pH in the anolyte facilitated (via sparging) removal of dissolved chlorine, which in turn mitigated the accumulation of elevated free chlorine levels at the completion of the EC treatment.

Overall, the bench scale testing provided a design basis for the field-scale EC system and confirmed that EC treatment of shower-based graywater for reuse was feasible.

### **5.3 SITE-SPECIFIC TREATABILITY STUDY DESIGN**

#### ***5.3.1 Study Objectives.***

The overall goal of the laboratory treatability study was to (1) determine the optimal level of low-pressure pre-filtration and (2) optimize the EC configuration in terms of COD removal, DBP formation, and energy consumption.

#### ***5.3.2 Treatability Study Methods***

##### **Graywater Collection and Filtration**

Experiments were performed using graywater collected in 5-gallon soda kegs from Camp Shelby (Hattiesburg, Mississippi). The graywater was collected from the collected effluent of shower and sink facilities; the graywater was collected under nitrogen headspace in the kegs. The soda kegs were shipped on ice to CDM Smith in Bellevue, Washington for analysis and testing, maintaining a temperature of 4°C. Upon arrival at Bellevue, the kegs were stored at approximately 4°C under nitrogen headspace and maintained by backfilling the kegs with nitrogen when samples were collected. The results of a characterization of the raw graywater are presented in Table 5.1.

The impact of low-pressure filtration on the graywater COD levels was evaluated. The COD level in the raw graywater was compared to the COD levels after vacuum filtration using 43 µm, 10 µm, and 1 µm filters. Results of this test are presented in Section 4.3.1. Before each electrochemical experiment, the graywater was vacuum filtered to the selected filtration level (1 µm) to prevent particulates from entering the EC cell and to reduce COD.

**Table 5.1. Characteristics of raw and filtered graywater.**

| Analyses                      | Unfiltered | 1 $\mu$ m Filtered |
|-------------------------------|------------|--------------------|
| Chloride (mg/L)               | 33         | 29                 |
| Sulfate (mg/L)                | 4.1        | 4.3                |
| Bromide (mg/L)                | <1.0       | < 1.0              |
| Nitrate (mg/L)                | <1.0       | 1.8                |
| Perchlorate ( $\mu$ g/L)      | <3.0       | < 3.0              |
| Chemical Oxygen Demand (mg/L) | 28 / 31    | 20 / 22            |
| Total Organic Carbon (mg/L)   | 5.3        | 4.7                |
| Conductivity ( $\mu$ S/cm)    | 380        | 390                |
| pH (standard units)           | 7.4        | 7                  |

### **Graywater Amendments**

The graywater used for this study was collected 2 days before a significant rain event, thus diluting the graywater. As shown in Table 4.1, COD and conductivity values in the raw graywater were approximately 2 and 10 times less than, respectively, what typically is expected in shower graywater sources (Hernández Leal et al., 2011; Leal et al., 2007). To allow for a more representative graywater composition for the laboratory testing (thus overcoming the precipitation-induced dilution of the graywater), the graywater was amended with 200 mg/L chloride as sodium chloride and 100 mg/L sulfate as sodium sulfate to increase graywater solution concentrations. This resulted in the electrical conductivity increasing to 1270  $\mu$ S/cm. To increase the graywater COD levels representative of undiluted shower graywater, the graywater was spiked with lactic acid (80 mg/L) (lactic acid solution 85% Sigma-Aldrich®) and Softsoap® hand soap (500 mg/L), increasing the COD to approximately 200 mg/L. The COD amendments were done in accordance with NSF/ANSI 350-2014 recipe for graywater. For the electrochemical experiments described in the following section, these amendments were added after filtering the graywater (1  $\mu$ m filter) and just prior to initiating the electrochemical testing.

### **EC Cell Configurations**

Microflow cells (ElectroCell North America, Inc.) parallel plate EC cells were used for all bench-scale testing. The experimental system is shown in Figure 5.1. The direct current power source was a Keithley 2220G-30-1 Dual-Output Programmable DC Power Supply. The EC cells each had wetted electrode areas (anode and cathode) of 10 cm<sup>2</sup>. In undivided cell configuration, the distance between the electrodes was 0.4 cm. In divided cell configuration, in which a Nafion 117 membrane was placed between the anodic and cathodic compartments, the distance between the electrodes was 0.8 cm. A Ti/IrO<sub>2</sub>-RuO<sub>2</sub> (1:1 IrO<sub>2</sub>:RuO<sub>2</sub> mass ratio) anode was used in all experiments except for one experiment where an SnO<sub>2</sub> anode was evaluated. Various cathodes were evaluated, including boron-doped diamond (BDD) on niobium (CONDIAS GmbH), stainless-steel, and solid graphite (G348). A summary of the experiments performed is provided in Table 5.2.

For the electrochemical experiments, the electrolyte solution consisted of 0.7 L of 1  $\mu\text{m}$  filtered and amended graywater, which was dispensed into a 1 L Erlenmeyer flask capped with a three-hole rubber stopper (allowing for influent, effluent, and headspace exchange tubes). The graywater was recirculated through the EC cell at 150 mL/min using a peristaltic pump. Batch treatment times ranged from 6 to 8 hours. For experiments performed using a divided EC cell configuration, two 0.7 L solutions were recirculated separately through the anolyte and catholyte chambers; one solution being the graywater and a second being an electrolyte solution of 500 mg/L  $\text{Na}_2\text{SO}_4$ . Divided EC cell experiments were evaluated using sequential anodic-cathodic treatment. The initial anolyte solution (graywater) was exchanged with the catholyte (electrolyte) halfway through the experiment such that the batch treatment time consisted of 4 hours anodic followed by 4 hours cathodic treatment.

All experiments were performed under constant current conditions, with current densities ranging from 0 (no current controls) to 25  $\text{mA}/\text{cm}^2$ . Sampling was performed to monitor COD, pH, active chlorine, anions, and disinfection byproducts (THMs, HAAs, and perchlorate) as a function of time. Voltage also was monitored. All experiments were performed in duplicate.

A separate set of experiments was performed to assess cathodic THM removal. These experiments were performed using the divided EC cell configuration. Filtered graywater was amended with 2000 ppb THMs in the catholyte and monitored hourly for THM loss. These experiments were performed at current densities of both 0 and 25  $\text{mA}/\text{cm}^2$ .

An additional divided cell experiment was performed in which the anolyte was sparged with air at approximately 70 mL/min during anodic treatment. This experiment was performed to take advantage of the low pH conditions that quickly developed in the anolyte, thereby facilitating the volatilization and removal of chlorine from the system. The subsequent impacts of this chlorine removal on the formation of disinfection byproducts were evaluated.

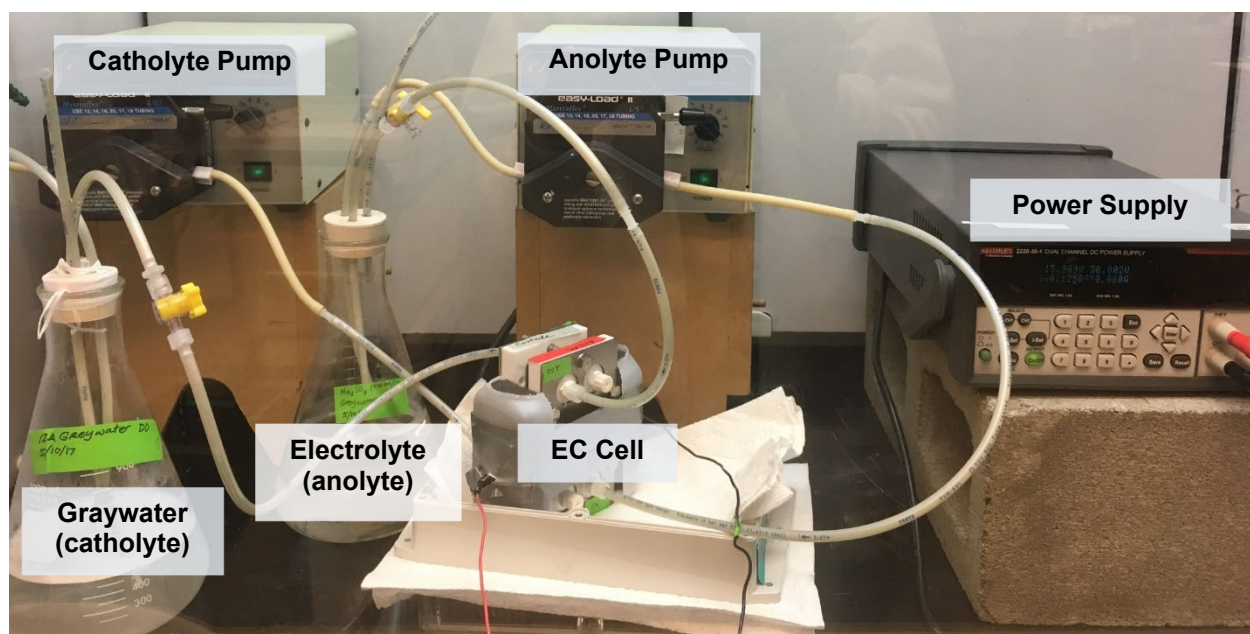


**Figure 5.1** Experimental system for the electrochemical experiments. The divided cell configuration is shown.

**Table 5.2. Summary of EC cell configurations evaluated**

| Undivided EC Cell Configuration                                 |                                                |
|-----------------------------------------------------------------|------------------------------------------------|
| Anode/Cathode Combination                                       | Current Densities Tested (mA/cm <sup>2</sup> ) |
| Ti/IrO <sub>2</sub> -RuO <sub>2</sub> / Stainless Steel         | 0, 0.3, 5, 12.5, 25                            |
| SnO <sub>2</sub> / Stainless Steel                              | 12.5                                           |
| Divided EC Cell Configuration (Sequential Anodic Cathodic)      |                                                |
| Anode/Cathode Combination                                       | Current Densities Tested (mA/cm <sup>2</sup> ) |
| Ti/IrO <sub>2</sub> -RuO <sub>2</sub> / Stainless Steel         | 5, 12.5                                        |
| Ti/IrO <sub>2</sub> -RuO <sub>2</sub> / Stainless Steel-Sparged | 12.5                                           |
| Ti/IrO <sub>2</sub> -RuO <sub>2</sub> / Graphite                | 12.5                                           |
| Ti/IrO <sub>2</sub> -RuO <sub>2</sub> /Boron Doped Diamond      | 12.5                                           |

### ***H<sub>2</sub> Capture and Analysis***

To capture H<sub>2</sub> evolving from the cathode in a divided cell, tests were carried out in Erlenmeyer flasks sealed with rubber stoppers with three holes for influent, effluent, and gas tubing. The gas line was connected to a Tedlar bag where gas was collected for analysis. The volume of air in the bag and headspace were recorded to account for dilution of the hydrogen gas being generated by the cathode. Samples were collected from the Tedlar bag. Samples were analyzed using the gas

chromatograph with a thermal conductivity detector (HP 5890A) (GC-TCD) with Restek ShinCarbon ST 80/100, 2 m x 2.0 mm ID (Restek Cat. #80486-810 or similar) column with a method developed in house at the Bellevue Research and Testing Laboratory. Samples were collected for analysis at time zero, midway through the experiment, and at the final time point and increased linearly to approximately 20% H<sub>2</sub> after 2 hours at an applied current density of 25 mA/cm<sup>2</sup>. Approximately 40 ml of hydrogen gas were generated in this screening test. The rate of hydrogen generation was be evaluated more closely during the field demonstration.

### ***Analytical Methods***

The graywater was monitored hourly during electrochemical treatment for pH, voltage, COD, THMs, anions, perchlorate, and HAA5. pH and temperature were measured using a Thermo Scientific™ Orion™ 9107BN Triode™ and voltage was monitored on the Kiethley 2220-30-1 Dual Channel DC Power Supply. COD was monitored using Low Range COD Digestion Vials Hach®, digested for 2 hours at 150°C in a Hach® COD reactor (model 45600), and analyzed with a DR-6000 Hach® spectrophotometer. Total THMs (chloroform, bromodichloromethane, dibromochloromethane, and bromoform) were analyzed by EPA methods 501.1, 524.2, and 601. A 5 mL ample was collected in 40 mL clear glass VOA vials with septa- and polytetrafluoroethylene-lined stir bars and quenched with 0.15 mg of ascorbic acid. THMs were quantified using a Tekmar 3000 Sample Concentrator purge-and-trap concentrator, an O-I Analytical Archon 4552 soil/water purge-and-trap autosampler, a Sigma K trap for Tekmar (Sigma-Aldrich Cat. #21066-4), an HP 6890A gas chromatograph, an Agilent 5973N mass spectrometer detector, a Restek Rtx®-VMS, 30 m, 0.25 mm ID, 1.4 µm film (Restek Cat.# 19915) gas chromatographic column, and analyzed with ChemStation version E.02.01.1177 software. Anions were analyzed by EPA method 300.1 (chloride, sulfate, bromide, nitrate) and perchlorate by EPA method 314.1. Both anions and perchlorate were quantified using a Dionex AS 140 autosampler, Thermoscientific AERS 300 suppressor, Dionex CD 20 conductivity detector, Dionex GP 40 gradient pump, and Dionex LC 20 enclosure and were analyzed with Chromeleon 6.8 SR10 software. The column and guard column used for anions were the IonPac AS18 4 × 250 mm and the IonPac AG18 4 × 50 mm, respectively, and the IonPac AS16 4 × 250 mm and IonPac AG16 4 × 50 mm column and guard column for perchlorate, respectively. HAA5 (monochloroacetic, dichloroacetic, trichloroacetic, bromoacetic, and dibromoacetic) were analyzed by EPA method 522.2 by an external lab (AmTest Laboratories in Kirkland, Washington). Total organic carbon (TOC), chlorate, and cations were measured at time zero and at the final collection point using Standard Methods 5310B, EPA method 300.1, and EPA method 6010, respectively. TOC was quantified using a Shimadzu TOC-VCSH and autosampler. Free chlorine was monitored using Hach® DPD Free Chlorine Reagent Powder Pillows and measured using a DR6000™ UV VIS Spectrophotometer, and peroxide was monitored using CHEMetrics Hydrogen Peroxide – Vacu-vials® Instrumental Kit and measured with a V-2000 photometer.

### 5.3.3. Treatability Study Results

#### 5.3.3.1. Filtration Results

Results from filtering pretests showed that filtering using a 10  $\mu\text{m}$  and 42  $\mu\text{m}$  filter did not result in any significant ( $> 10\%$ ) removal of COD. However, use of a 1  $\mu\text{m}$  filter resulted in a 30% decrease in COD (Figure 4.2). Thus, 1  $\mu\text{m}$  filtration was selected for the filtration level. Filtration substantially below 1  $\mu\text{m}$  likely would require increased energy and maintenance for field-scale application. Therefore, it was not investigated in this study. In accordance with the findings of this pretest, all subsequent electrochemical experiments were performed using 1  $\mu\text{m}$  vacuum filtered graywater.

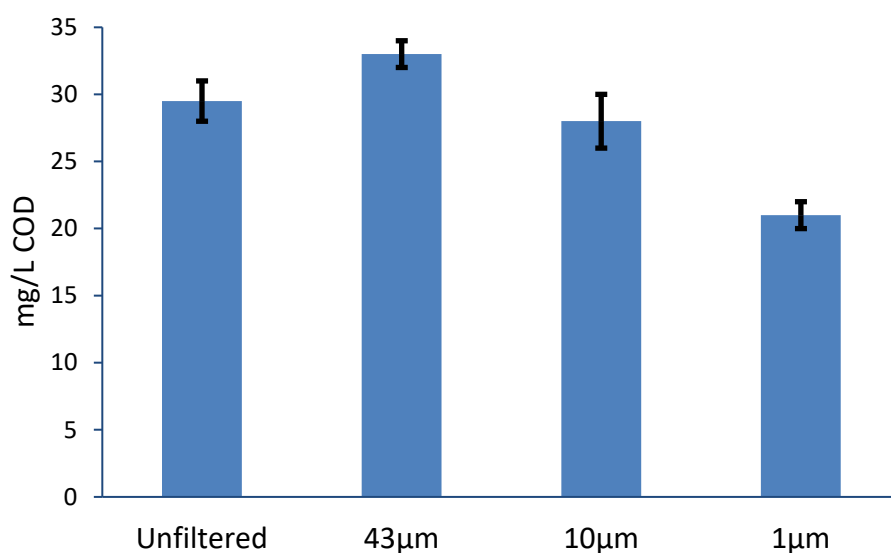


**Figure 5.2. Filtering with a 1  $\mu\text{m}$  filter results in a 30% loss in COD. The error bars represent the standard deviation.**

#### Undivided Cell Experiments

##### *COD Removal as a Function of Current Density*

COD removal increased with time and current density in the undivided cells with the  $\text{Ti}/\text{IrO}_2\text{-RuO}_2$  anodes and stainless steel cathodes. Negligible COD removal was observed at 0 and 5  $\text{mA}/\text{cm}^2$ , approximately 40% removal was observed at 12.5  $\text{mA}/\text{cm}^2$ , and nearly 60% removal at 25  $\text{mA}/\text{cm}^2$  (Fig 4.3). COD removal was linear with respect to time for the 12.5 and 25  $\text{mA}/\text{cm}^2$  current densities. The energy required for a 50% COD removal was approximately 17  $\text{W-h L}^{-1}$ . This value is in reasonable agreement for energy needed for 50% COD removal (29  $\text{W-h L}^{-1}$ ) in high salinity leachate (Anglada et al., 2009); the lower energy demand observed by Urtiaga et al. was likely because the electrical conductivity was substantially greater compared to the graywater

used in this current study. The coulombic efficiency for COD removal, assuming 2 electrons per mole of COD, was 3% (Martínez-Huitle and Ferro, 2006).

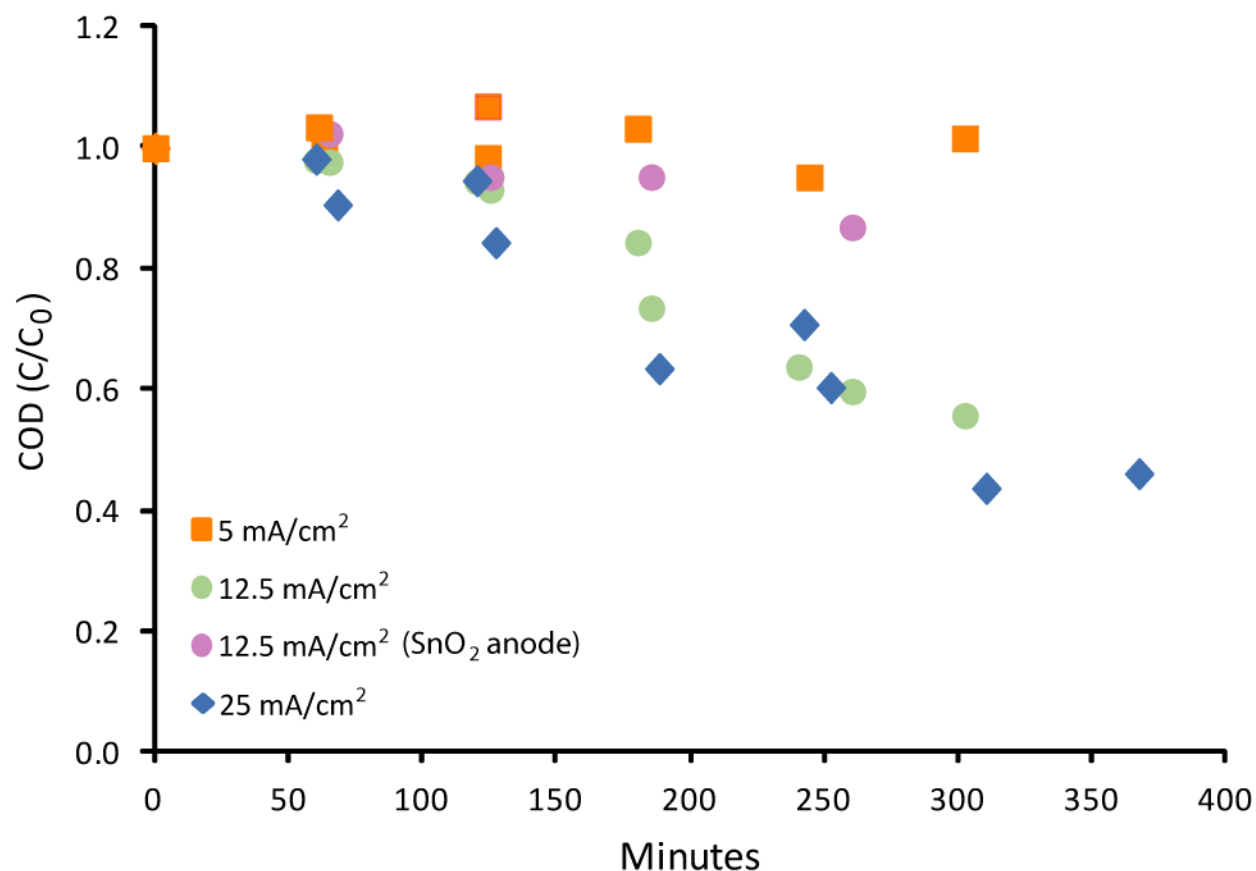


**Figure 5.3. Removal of COD as a function of time for the various current densities tested in the undivided cell configuration. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and stainless steel cathode, except where noted. Cell voltages ranged from 0 to 10 V. pH generally varied between 6.8 and 8.1, with gradual increases observed during treatment.**

#### *Electrochemical Disinfection Byproduct Generation*

THM, HAA5, and perchlorate generation were a function of the applied charge, as shown in Figures 4.4 to 4.6. The generation of these DBPs were accompanied by elevated levels of free chlorine (in excess of 200 mg/L at the end of the experiments performed at 12.5 and 25 mA/cm<sup>2</sup>); previous studies have shown the presence of elevated free chlorine levels facilitate DBP formation (Hong et al., 2009). For THMs, the rate of formation was not proportional to the applied current density ranging from approximately 22 µg A<sup>-1</sup> min<sup>-1</sup> at 25 mA/cm<sup>2</sup> to approximately 5.5 µg A<sup>-1</sup> min<sup>-1</sup> at 5 mA/cm<sup>2</sup>. Thus, THM generation was not proportional to the specific charge (A-min), which is consistent with previous observations (Garcia-Segura et al., 2015), and highlights the complex nature of THM formation in electrochemical systems.

In contrast, although scatter in the data prohibits precise quantitative analysis, generation of HAA5 was proportional to the applied current density (Figure 5.5). The approximate rate of HAA5 generation was  $4 \mu\text{g A}^{-1} \text{min}^{-1}$ . This rate of HAA5 generation is in reasonable agreement with HAA5 generation observed during electrochemical disinfection of surface water (Schaefer et al., 2015).

Perchlorate generation was only observed at elevated current densities (12.5 and 25  $\text{mA/cm}^2$ ); perchlorate was below the analytical detection limit of  $1 \mu\text{g/L}$  in the other experimental conditions. A lag was observed prior to any measurable generation of perchlorate. This lag likely was due to initial generation of lesser oxidized products such as chlorate (Azizi et al., 2011).

With respect to the composition of the THMs, chloroform generation in the undivided cells was significantly higher than any other THM (*t* Tests;  $p=0.05$ ). After chloroform, bromodichloromethane (BDCM) was the second most abundant THM. Bromoform and chlorodibromomethane were only present in trace amounts, accounting for 0.5% of the molar fraction of the total THMs. The abundance of the chlorine-rich THMs is consistent with the presence of elevated free chlorine levels and bromide levels near the analytical detection limit of  $0.001 \text{ mg/L}$ .

The molar ratio of chloroform to BDCM increased over time for each of the current densities tested (Figure 5.7). This observation is likely due to the removal of bromide over time via electrochemical treatment, which has been previously observed to limit brominated THM formation (Schaefer et al., 2017).

For HAA5, and consistent with the THM composition, monochloroacetic acid, dichloroacetic acid, and trichloroacetic acid were formed, but the brominated HAAs remained below the analytical detection limit of 20 ppb. Because of volume constraints, detection limits for HAA5 were higher than expected. In contrast with THM results, there was no clear ratio trends with any of the HAAs over the course of the experiment.

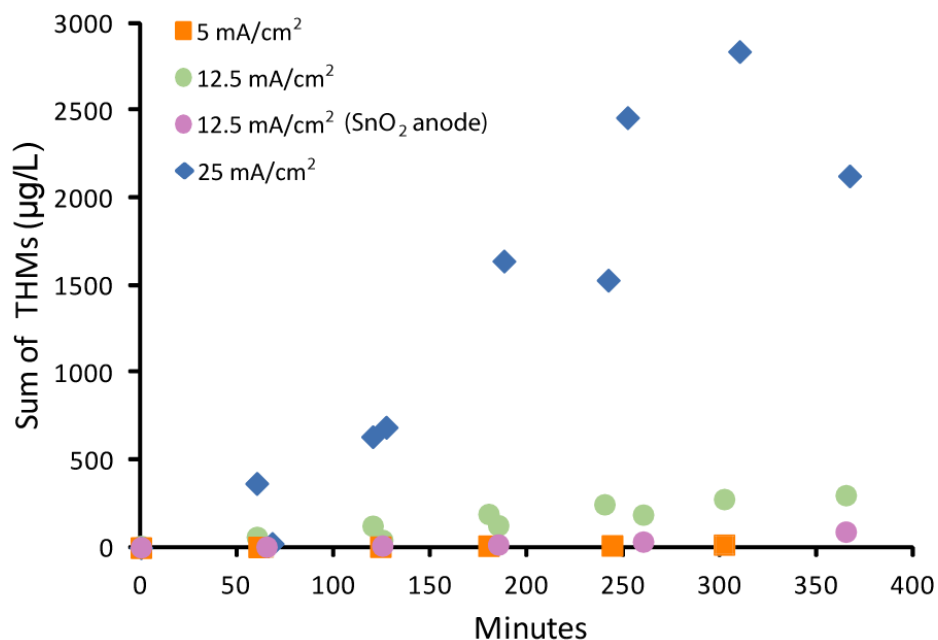


**Figure 5.4. THM generation in undivided EC cells. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and stainless steel cathode.**

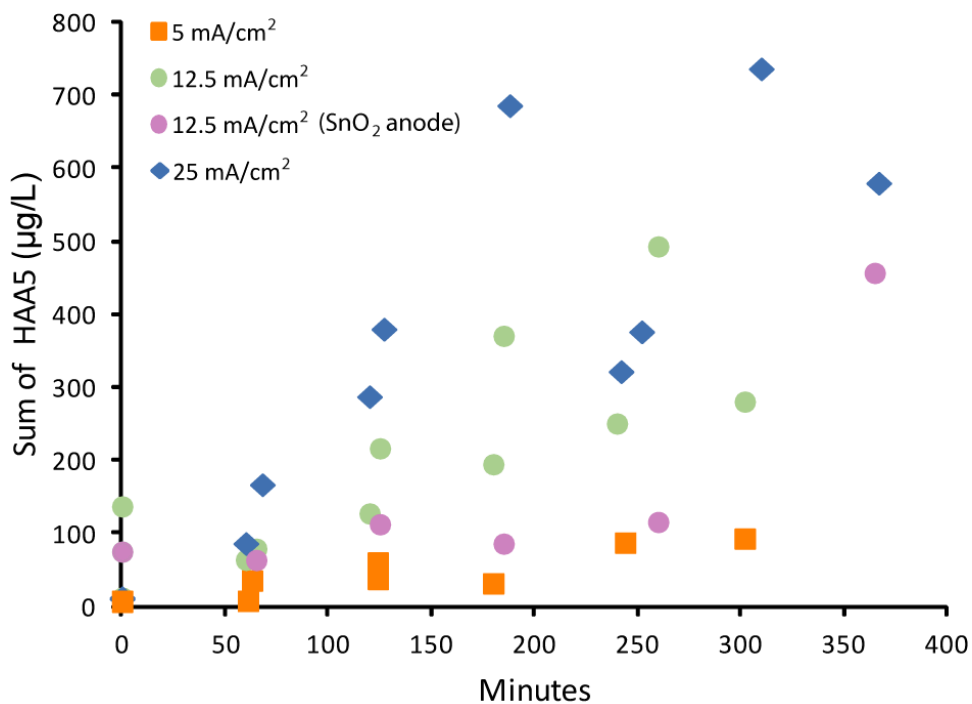


**Figure 5.5. HAA5 generation in undivided EC cells. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and stainless steel cathode.**

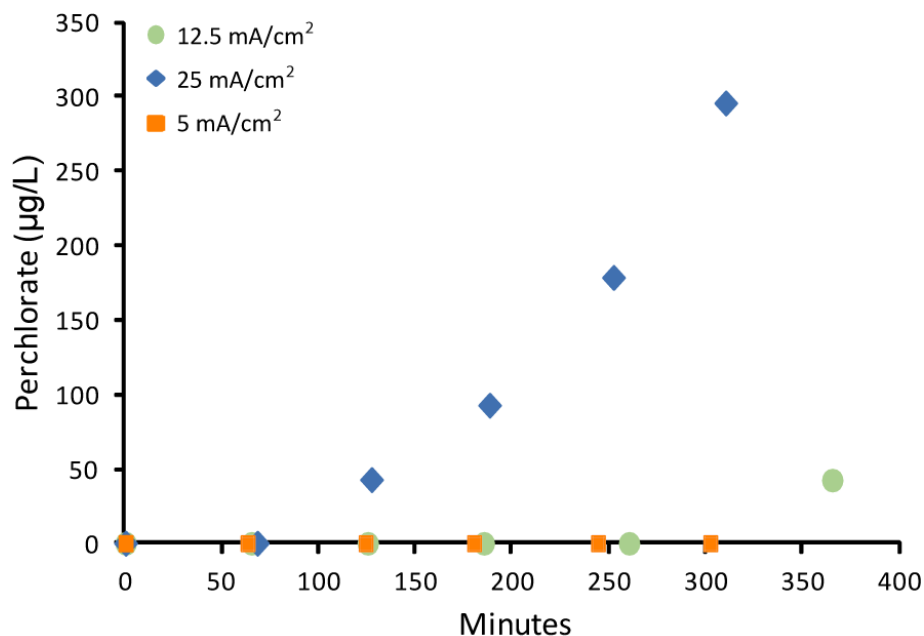


**Figure 5.6. Perchlorate generation in undivided EC cells. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and stainless steel cathode.**

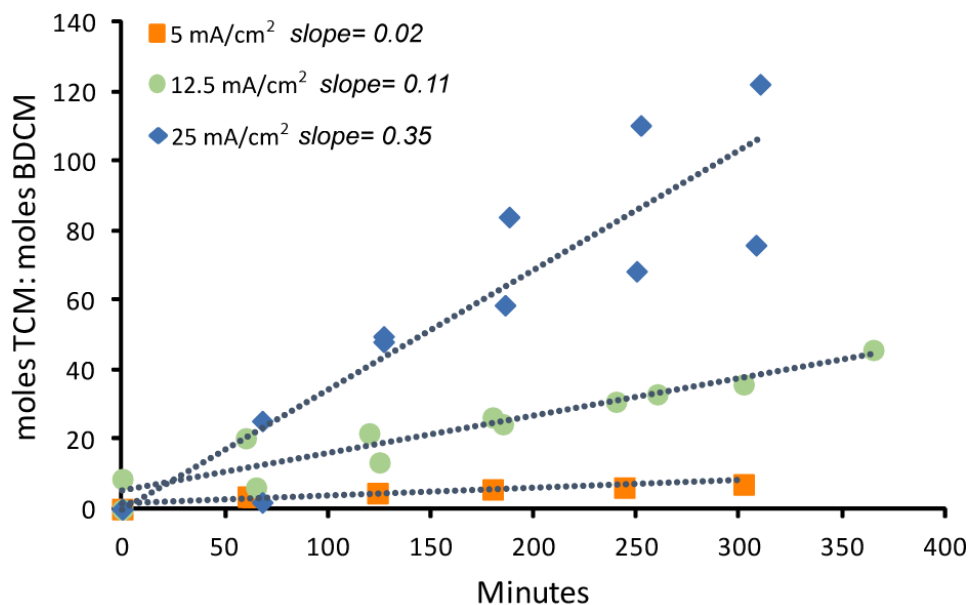


**Figure 5.7. Change in molar chloroform: BDCM ratios for three current densities tested in the undivided EC cell configuration. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and stainless steel cathode.**

Chloride concentrations decreased with the current density, where a 27%, 32%, and 43% decrease in chloride was observed at 5, 12.5, and 25 mA/cm<sup>2</sup>, respectively. The majority of oxidized

chlorine remained as free chlorine, 83% and 79% (molar basis) in the 12.5 mA/cm<sup>2</sup> and 25 mA/cm<sup>2</sup> experiments respectively. Only trace amounts of chloride yielded DBP formation (2 and 3% in the 12.5 mA/cm<sup>2</sup> and 25 mA/cm<sup>2</sup> experiments respectively).

#### *SnO<sub>2</sub> Anode Comparison*

An SnO<sub>2</sub> anode was compared to the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode at 12.5 mA/cm<sup>2</sup>. Employing a SnO<sub>2</sub> anode resulted in lower rates of THM and HAA5 generation (approximately half), but COD removal rates were slightly lower compared to the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode. Because of the lower rate of COD removal, the SnO<sub>2</sub> anode was not considered for further testing.

### **5.3.3 Divided Cell Experiments**

#### *COD Removal*

COD removal rates for the sequential anodic-cathodic divided EC cell experiments performed at 12.5mA/cm<sup>2</sup> are shown for each cathode material in Figure 5.8. Overall, in the absence of sparging, COD removal was the lowest using the stainless steel cathode and greatest using the BDD and graphite cathodes. Consistent with previous studies comparing anodic and cathodic COD removal from wastewater (Wang et al., 2009), COD removal was greater in the anolyte than in the catholyte, as oxidation of COD in the anolyte likely was facilitated by both hydroxyl radical generation, oxidation via oxidized chlorine species, and /or direct electron transfer (Bagastyo et al., 2011, 2013). Removal of COD in the catholyte can occur via generation of hydrogen peroxide and/or reduction at the cathode (Rao and Venkatarangaiah, 2014). Measured hydrogen peroxide levels during cathodic treatment were typically below 1 mg/L, so it is unlikely that cathodic hydrogen peroxide generation played a dominant role in COD removal. The relatively poor anodic removal of COD when using the stainless steel cathode without sparging (panel C in Figure 5.8) is not readily explained.

Air sparging in the anolyte solution resulted in an increase in the anodic COD removal rate of nearly 3 (panels C and D in Figure 5.8) This increase in COD removal in the anolyte may have been due to enhanced removal of competing electron donors such as Cl<sub>2</sub>. Alternately, sparging could have removed some volatile intermediate oxidation products from the graywater COD.

The net energy required for a 50% COD removal in the divided EC cell configuration (sequential anodic-cathodic) for both the BDD and graphite cathodes was approximately 13 W-h L<sup>-1</sup>, which is 24% less than what was observed in the undivided cell configuration. For the stainless steel (unsparged), the energy demand for 50% COD removal was approximately 20 W-h L<sup>-1</sup>, which is slightly greater than what was observed in the undivided configuration. On the cathodic side, the improved energy efficiency for the BDD and graphite cathodes likely is due to direct cathodic reduction of the organics present in the graywater. The differences in COD removal rate in the anodic compartment among the three cathodes tested (particularly for the stainless steel) is not readily explained, although differences in anodic treatment have been previously observed to be dependent upon the cathode materials in divided EC cells (Rao and Venkatarangaiah, 2014). The measured cell voltage and pH were not measurably impacted by the anode material, thereby

eliminating these parameters as an explanation. One possible explanation is that the anode material impacted migration of cations across the membrane (via transformation at the cathode surface), thereby indirectly affecting anodic treatment.

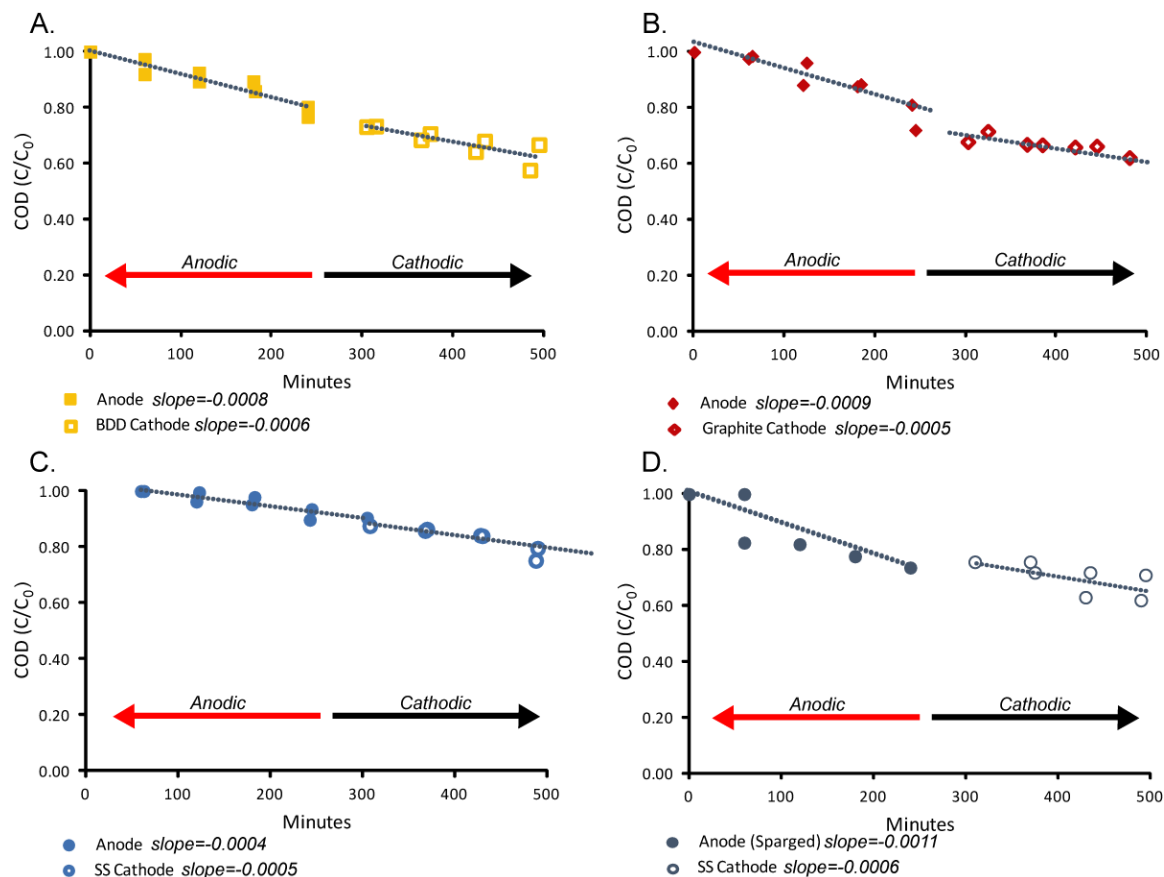


**Figure 5.8. COD removal in the divided EC cell configuration as a function of time using various cathode materials. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and were performed in duplicate. The applied current density was 12.5 mA/cm<sup>2</sup>, and the cell voltage was approximately 11V. A = BDD cathode, B = graphite cathode, C = stainless steel cathode, and D = stainless steel cathode while air sparging in the anolyte solution.**

### *Electrochemical Disinfection Byproduct Generation*

Generation of free chlorine and chlorinated byproducts were substantially different from observations made in the undivided EC cell experiments. Figure 5.9 shows, for all three anodes tested, free chlorine concentrations increased in the anolyte, then remained constant or slightly declined in the catholyte. Free chlorine levels were similar to those observed under undivided conditions, but the pH extremes ensured the speciation of the oxidized chlorine was different, as the free chlorine in the anode (pH = 2.2) was likely present as Cl<sub>2</sub> and as hypochlorite in the catholyte (pH = 12). Free chlorine levels were slightly greater when using the BDD and graphite

cathodes compared to the stainless steel. This increased rate of chloride oxidation using the BDD and graphite anodes is consistent with the removal of COD, indicating that a greater rate of oxidation was observed using these cathode materials compared to the stainless steel. ***In the presence of anodic sparging, free chlorine levels were substantially diminished (less than 2 mg/L free chlorine by the end of cathodic treatment) due to removal of  $Cl_2$  in the anolyte.***

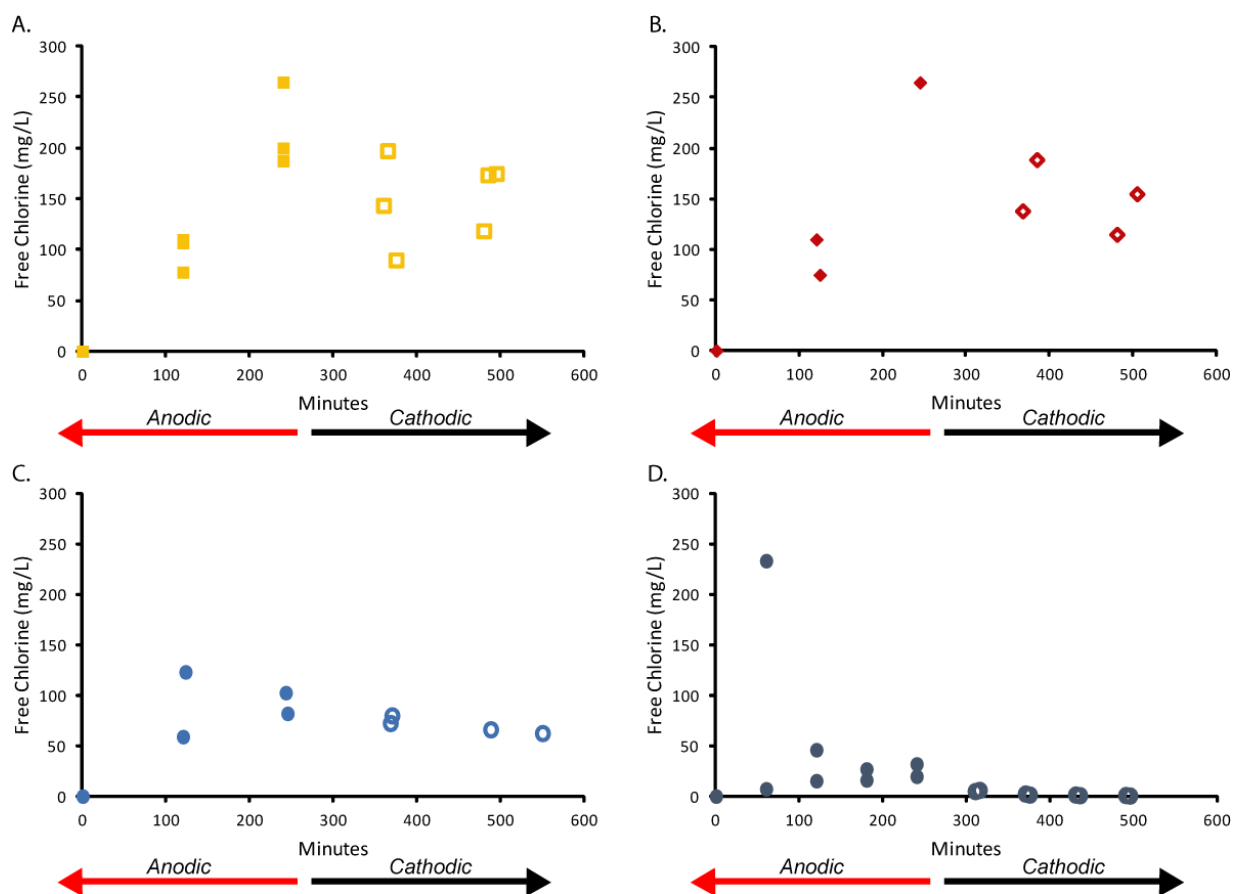


**Figure 5.9. Free chlorine generation in the divided EC cell configuration as a function of time using various cathode materials. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and were performed in duplicate. The applied current density was 12.5 mA/cm<sup>2</sup>, and the cell voltage was approximately 11V. A = BDD cathode, B = graphite cathode, C = stainless steel cathode, and D = stainless steel cathode while air sparging in the anolyte solution.**

THM results for each of the three cathode materials and HAA5 for the BDD cathode are shown in Figures 4.10 and 4.11. For each cathode material, THMs did not exhibit any appreciable accumulation in the anolyte. Previous studies have shown HAA formation is favored at low pH (Bagastyo et al., 2011; Liang and Singer, 2003; Singer, 1994), which likely explains why HAA5 generation was observed, but not THMs, in the anolyte. The rate of HAA5 formation in the anolyte is similar (within a factor of 2) to that observed for the undivided configuration.

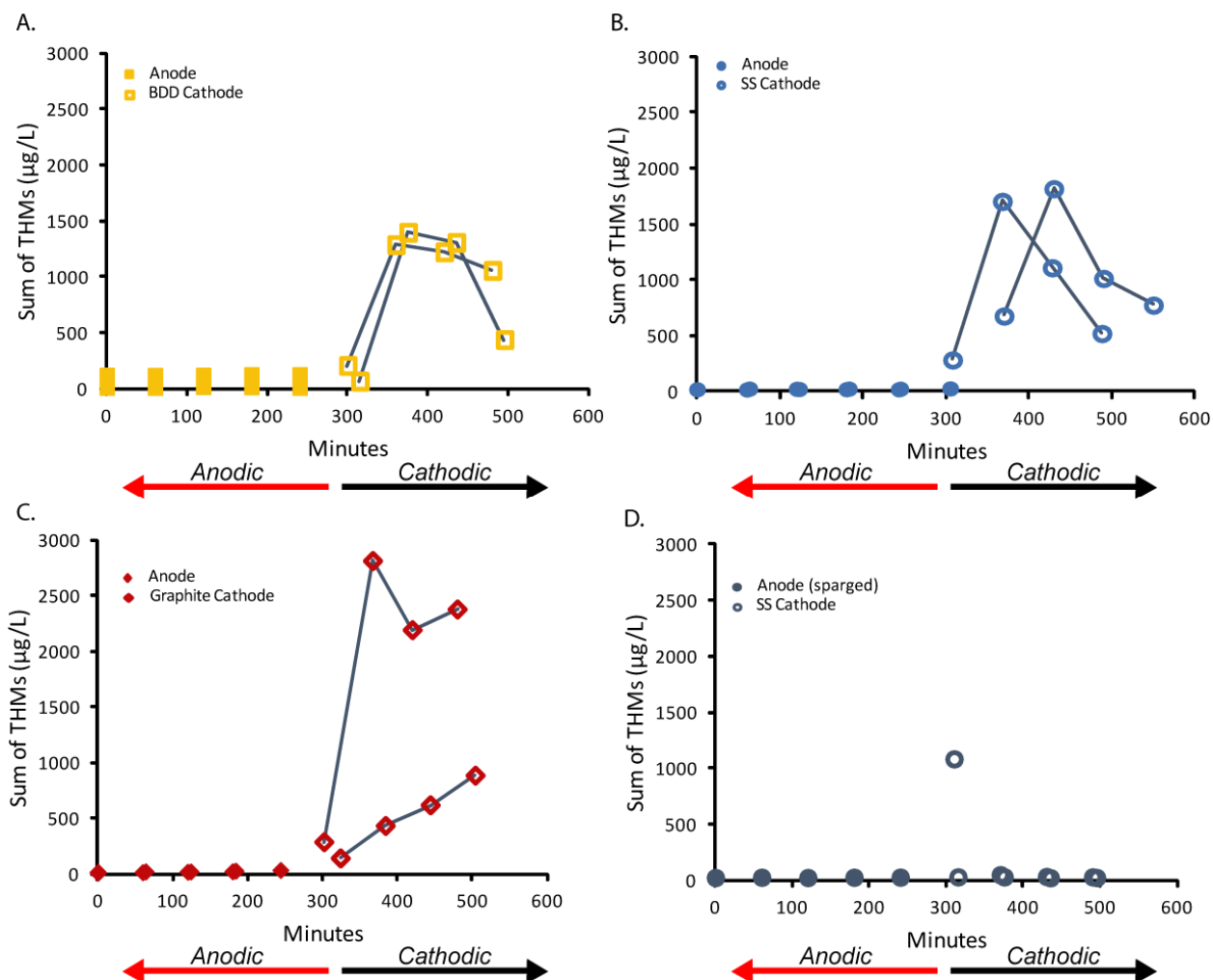


**Figure 5.10. THM generation, subsequent cathodic transformation of THMs, and free chlorine in divided cells where THM removal varies with cathodic materials. All experiments were performed using the Ti/IrO<sub>2</sub>-RuO<sub>2</sub> anode and were performed in duplicate. The applied current density was 12.5 mA/cm<sup>2</sup>, and the cell voltage was approximately 11V. A = BDD cathode, B = graphite cathode, C = stainless steel cathode, and D = stainless steel cathode while air sparging in the anolyte solution.**

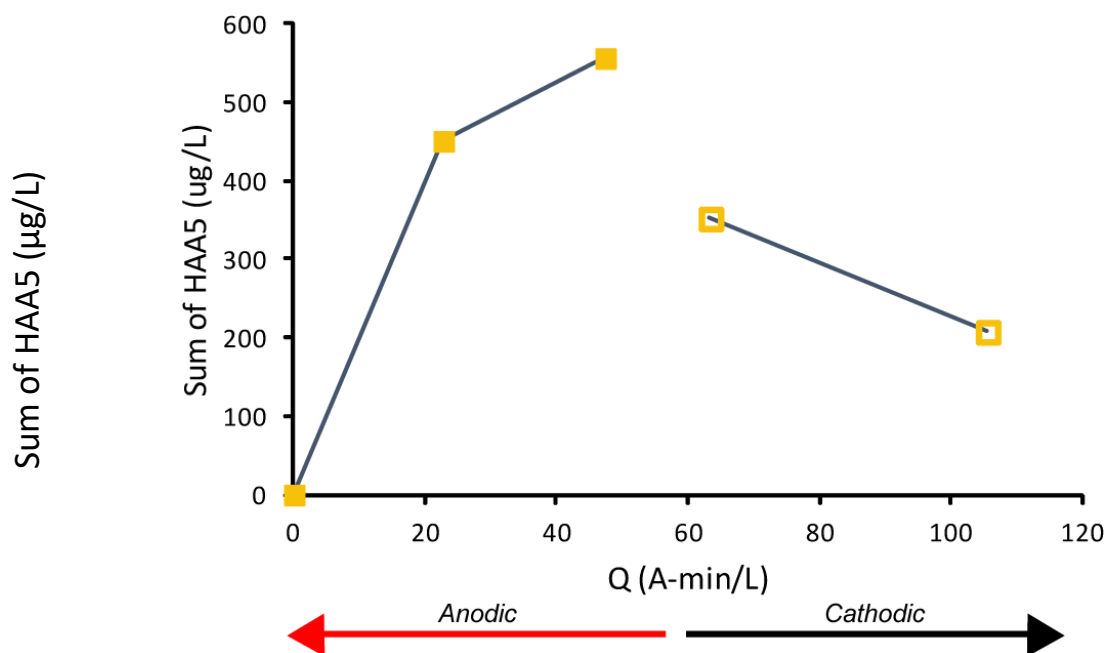


**Figure 5.11. HAA5 loss under higher pH. HAA5 were not monitored in the stainless steel and graphite divided cell experiments.**

Once the anolyte solution was transferred to the catholyte and cathodic treatment was initiated, THM concentrations greatly increased to levels that were substantially greater than those observed in the 12.5 mA/cm<sup>2</sup> condition for the undivided configuration. This result suggests that pH had a great impact on the extent of THM generation, and increasing the pH resulted in rapid formation of THMs. The initial increase in THMs upon transfer to the catholyte was similar for all three cathodes, suggesting THM formation was driven by bulk solution chemistry and the transformation of dissolved chlorine and hypochlorous acid to hypochlorite. For the sparged condition (Panel D in Figure 5.10), final THM generation was near the analytical detection limit of 20 µg/L, likely because the free chlorine levels were greatly diminished.

The molar ratios of the individual THMs and HAA5 in the anolyte were generally consistent with those observed for the undivided electrochemical experiments. These ratios remained consistent in the catholyte, even as THMs were being removed. For both the BDD and stainless steel cathodes, THM levels began to decline in the anolyte after the initial THM spike. This reduction was likely due to cathodic reduction at the anode (Esclapez et al., 2013; Garcia-Segura et al., 2015). In congruence with this reduction in THMs, chloride concentrations increased by 12% in the BDD cathode experiments and 9% in the Graphite cathode experiments between the last time point at the anode and the final time point at the cathode. Removal of HAA5 in the catholyte also was observed when using a BDD cathode. Thus, sequential anodic-cathodic treatment provides a potential approach for mitigating the net generation of DBPs. ***Perchlorate generation was not observed, as perchlorate remained below the analytical detection limit of 1 µg/L.***

The stainless steel cathode had to be replaced following duplicate experiments, as this cathode exhibited significant corrosion and was prevented from use in additional experiments (Figure 5.12).



**Figure 5.12. Corroded stainless steel cathode from the divided EC cell experiments.**

#### *Additional Assessment of Cathodic THM Removal*

To further evaluate the cathodic rate of THM removal, THM-spiked graywater (not subjected to anodic pre-treatment) was cathodically treated to verify and quantify THM removal. Results are shown in Figure 5.13. THM losses with zero applied current are likely due to volatilization and/or sorption. The difference between the control (no applied current) and where current was applied is assumed to be due to cathodic treatment.

Regressed first-order rate constants for the individual THMs are shown in Table 5.3. Results show THM removal is reasonably described by first-order kinetics. THM removal is generally greater for the stainless steel cathode than for the BDD cathode. For the stainless steel cathode, THM removal decreased with increasing Br content; this trend was not observed for the BDD cathode.

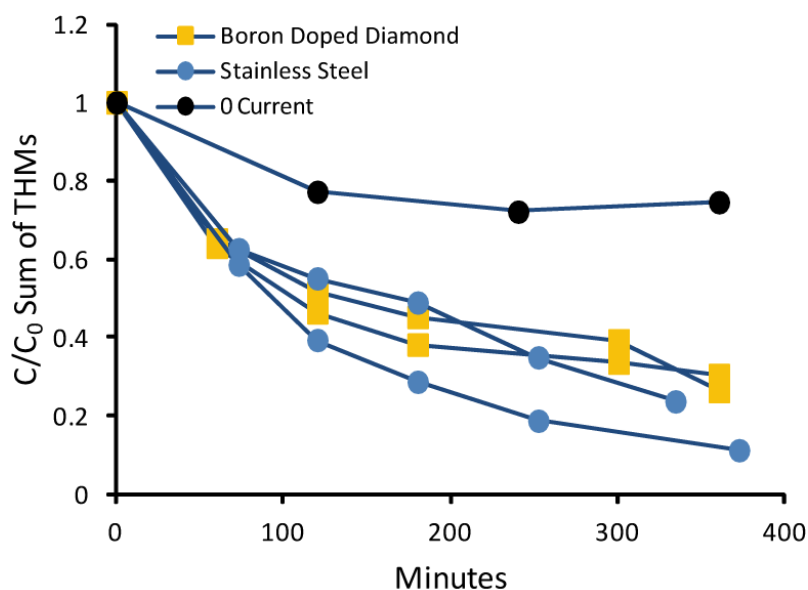


**Figure 5.13. THM transformation attributable to volatilization (black circles) versus cathodic destruction at 25 mA/cm<sup>2</sup> using boron-doped diamond (yellow squares) and stainless steel cathodes (blue circles).**

**Table 5.3. Regressed first-order rate constants for cathodic removal of THMs in graywater at 12.5 mA/cm<sup>2</sup>. Plus-minus (±) values indicate the 95% confidence intervals. Regressions are normalized to the losses observed in the no current controls. TCE = trichloromethane, BDCM = bromodichloromethane, DBCM = dibromochloromethane, and TBM – tribromomethane.**

|         | TCM                                |                | BDCM                               |                | DBCM                               |                | TBM                                |                |
|---------|------------------------------------|----------------|------------------------------------|----------------|------------------------------------|----------------|------------------------------------|----------------|
| Cathode | Rate Constant (min <sup>-1</sup> ) | R <sup>2</sup> | Rate Constant (min <sup>-1</sup> ) | R <sup>2</sup> | Rate Constant (min <sup>-1</sup> ) | R <sup>2</sup> | Rate Constant (min <sup>-1</sup> ) | R <sup>2</sup> |
| SS      | 0.0069 ± 0.00052                   | 0.96           | 0.0051 ± 0.00044                   | 0.94           | 0.0037 ± 0.00036                   | 0.92           | 0.0030 ± 0.00028                   | 0.92           |
| BDD     | 0.0027 ± 0.00037                   | 0.80           | 0.0035 ± 0.00035                   | 0.89           | 0.0031 ± 0.00032                   | 0.88           | 0.0030 ± 0.00031                   | 0.87           |

## 5.4 DESIGN AND LAYOUT OF TECHNOLOGY COMPONENTS

The demonstration was performed at the Presidio of Monterey, outside building 627B. The pilot system treated shower discharge from a bathroom in building 627B. The graywater was treated in 30 gallon batches, lasting between 5 and 8 hours. The treatment system consists of pre-filtration, electrochemical treatment, chlorine reduction, and pH adjustment. The specifics of the system are described in the following sections, and a process and instrumentation diagram is shown in Figure 5.1

### 5.4.1 Design of the EC System

Our design and operation plan for the system and serves as our basis, modifications may be made to facilitate improved operation and evaluation of the system, thus the intent of the following is to guide the construction, programming, and installation.

The pilot system operates in a continuous batch sequence. At the start of a cycle, a valve on the influent line opens to allow graywater to flow in the Aqua2use GWDD graywater filter which removes hair and large particles. An internal pump in the Aqua2use GWDD graywater filter conveys filtered water consecutively through a 50 micron and a 1 micron pressure filter and into tank T-100. During the demonstration phase of the project, an ion exchange system was installed ahead of the filters to remove hardness due to scaling issues inside the cell. Float 1 is activated when the tank is full, and the valve closes.

The power supply turns on to apply electric current to the electrochemical cell. The electrochemical cell is in a divided configuration with a total cathode area of 1000 cm<sup>2</sup> and total anode area of 1000 cm<sup>2</sup>. Boron-doped diamond electrodes served as cathodes, and Ti/IrO<sub>2</sub>-RuO<sub>2</sub> electrodes served as anodes. Nafion 117 membranes divided each cell.

Graywater is recirculated through anodic and cathodic portions of the electrochemical cell at approximately 1 L/min. The system was designed so that, during that time, if the free chlorine concentration in tank T-100 is greater than 50 mg/L, the tank is sparged with air until the free chlorine is less than 25 mg/L. However, the online chlorine analyzer failed during the demonstration phase, so this step did not occur. Recirculation continues until the COD in tank T-100 is below 50 mg/L. The COD analyzer also had continual issues, so recirculation was set on a timer instead. The duration was between 4 and 6 hours, depending on the particular cycle. Treated water was pumped to the finished water tank and pH is adjusted with acid until the pH is less than 8.5. At the same time graywater and electrolyte are recirculated through the electrochemical cell while it operates for several seconds in reverse polarity mode to mitigate carbonate scaling. When these functions are complete, the next batch starts. The system also has programmed sequences for shutdown (when the entire system is drained) and cleaning (when acid is recirculated through the cell).

The system was designed for independent operation. All of control scheme functions can be initiated remotely through a cellular connection at the PLC. The system process and instrumentation diagram (Figure 5.1). A photograph of the pilot system is shown in Figure 5.2 and the design criteria for the pilot system are in Table 5.1.

**Table 5.4. Pilot Design Criteria**

| Parameter   | Value | Units      |
|-------------|-------|------------|
| Flow rate   | 1     | L/min      |
| Max Voltage | 12.5  | V          |
| Max Amps    | 40    | Amp        |
| Power       | 375   | W          |
| COD         | <10   | mg/L       |
| E. coli     | <1    | cfu/100 mL |
| HAA5        | <60   | µg/L       |
| TTHM        | <80   | µg/L       |
| Perchlorate | <5    | µg/L       |

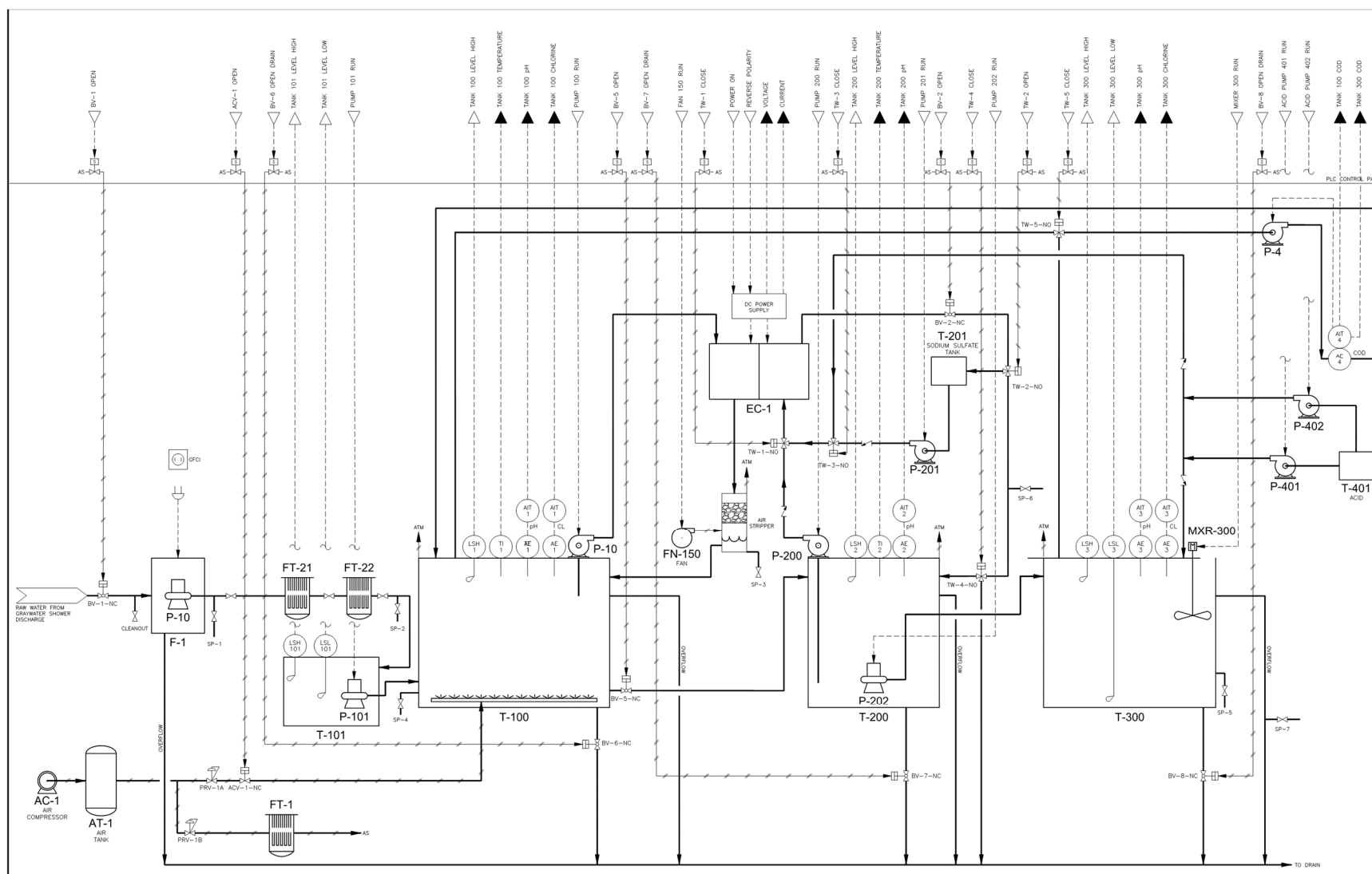


**Figure 5.14. Process and Instrumentation Diagram**

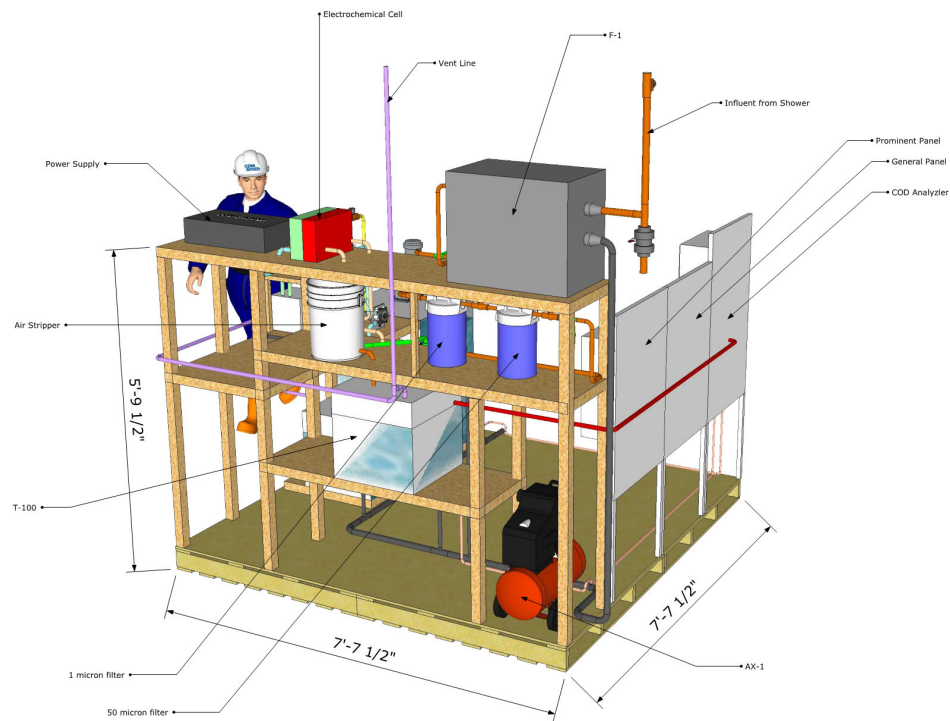


**Figure 5.15. 3-D Rendering of Pilot System**

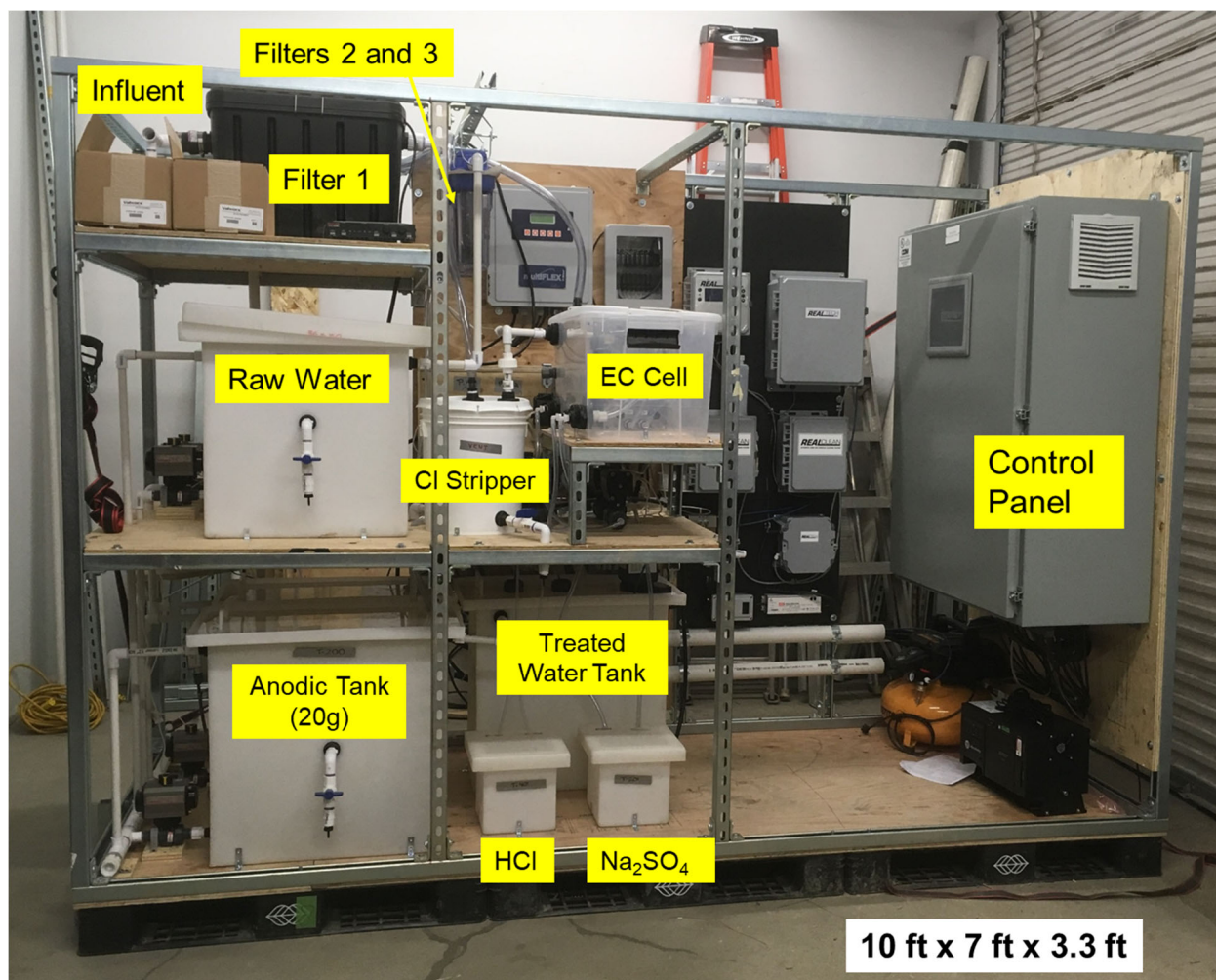


**Figure 5.16. Photograph of Pilot System**

## 5.5 FIELD TESTING

Field activities included system construction, installation, start-up testing, operations, and demobilization. The project schedule is summarized in the Gantt chart shown in Figure 5.17. It is noted that the schedule is preliminary and may be modified pending results observed in the field. Details of each of the 4 field activities are provided in the following sub-sections.

| ID | Task Name             | Start     | Finish    | Duration (d) | Q3 17 |     |     | Q4 17 |     |     | Q1 18 |     |     | Q2 18 |     |     | Q3 18 |     |     | Q4 18 |     |     | Q1 19 |     |     | Q2 19 |     |     |
|----|-----------------------|-----------|-----------|--------------|-------|-----|-----|-------|-----|-----|-------|-----|-----|-------|-----|-----|-------|-----|-----|-------|-----|-----|-------|-----|-----|-------|-----|-----|
|    |                       |           |           |              | SEP   | OCT | NOV | DEC   | JAN | FEB | MAR   | APR | MAY | JUN   | JUL | AUG | SEP   | OCT | NOV | DEC   | JAN | FEB | MAR   | APR | MAY | JUN   | JUL | AUG |
| 1  | Pilot Design          | 1-Sep-17  | 14-Dec-17 | 104          |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |
| 2  | Equipment Procurement | 31-Dec-17 | 9-Feb-18  | 40           |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |
| 3  | Pilot Construction    | 16-Apr-18 | 27-Apr-18 | 11           |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |
| 4  | Pilot Installation    | 22-May-18 | 25-May-18 | 3            |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |
| 5  | Operation             | 28-May-18 | 10-Jan-19 | 227          |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |
| 6  | Demobilization        | 13-May-19 | 15-May-19 | 2            |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |       |     |     |

**Figure 5.17: Project Schedule**

### ***5.5.1 System construction***

The pilot system was constructed in Denver, CO at a CDM Smith facility. Construction was performed by CDM Constructors, Inc (CCI) and overseen by Josh Goldman. The control panel constructed in Rancho Cucamonga, CA was shipped to Denver for installation on the pilot skid. An integrator was onsite to program the PLC and assisted with testing. The pilot system construction start date was February 12<sup>st</sup>, 2018.

The completed system was tested for water tightness, controls, and remote communications. All instrumentation was calibrated and checked. The complete skid was shipped by truck to the Presidio of Monterey.

### ***5.5.2 System installation***

Prior to the arrival of the pilot system, electrical and plumbing subcontractors prepared the site. An empty metal ISO shipping container installed on a wooden foundation as near to the showers as possible on the side of Building 627B. A 2" PVC pipe tied in-line to redirect the shower discharge into the pilot system container with a discharge line to the sewer connection nearby. A temporary electrical service line was extended from Building 627B to power the pilot system. The pilot skid was installed inside the metal container and connected to electrical service, the influent shower water and the sewer discharge line.

### ***5.5.3 Start-Up Testing***

With installation completed, start-up testing lasted 10 days. Each control scheme was tested, and remote communications verified. All subsystems were tested with graywater and operations of each unit process verified including filtration, COD removal, and disinfection. The overall control scheme (recirculation time, sparging time, pH adjustment time) was verified and applied to the PLC controls.

### ***5.5.4 System Operation & Monitoring***

The system was operated in continuous batch configuration, with treatment times for each batch varying between 6 and 8 hours. All operations occurred independently, and all sequences were controlled remotely. Physical graywater sampling events occurred bi-monthly. The system operated 24 hours per day and 7 days per week unless sampling or maintenance was conducted or there was no water is available from the showers. Project staff conducted 2 to 3 site visits during the operational period to make any required adjustments to the system.

### ***5.5.5 Demobilization***

The treatment system was taken disassembled, and the building plumbing was be returned pre-demonstration operation.

## 5.6 SAMPLING METHODS

Graywater sampling was conducted to assess the overall performance of the EC system with respect to graywater treatment, and system performance data from on-line instrumentation was collected and stored. In addition, the rate of cathodic hydrogen generation was to be estimated, as well as the overall energy consumption of the system.

Details of the sampling and monitoring were performed as part of this demonstration are described in the sections below.

### 5.6.1 Graywater System Sampling Locations and Analyses

Graywater samples were collected at sequential points along the treatment process and test for several analytes over the duration of the demonstration, including both the start-up and operational periods. Samples were collected from the following locations, shown in Figure 5.5.

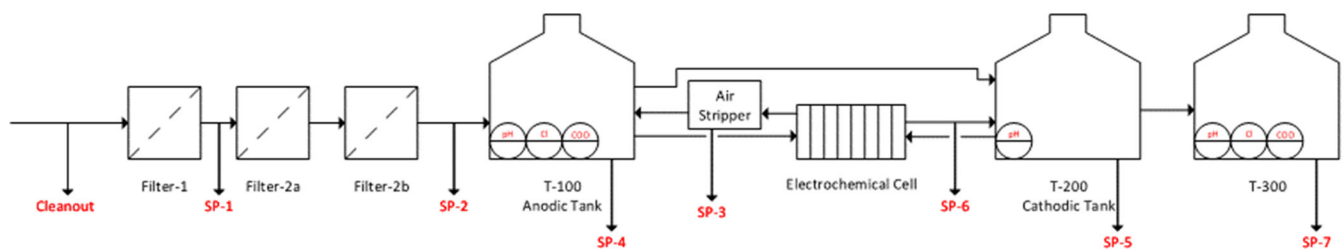


**Figure 5.18. Process Flow Diagram with Sample Locations**

- Cleanout: “raw” graywater collected prior to any treatment
- SP1: graywater collected after the pre-filter
- SP2: graywater collected after the 50  $\mu\text{m}$  and 1  $\mu\text{m}$  filters
- SP3: anodically treated graywater (grab)
- SP4: anodically treated graywater (composite)
- SP5: cathodically treated graywater (composite)
- SP6: cathodically treated graywater (grab)
- SP7: finished water

The frequency of sampling and analysis is shown in Table 5.2. The locations and frequencies listed may be modified during the demonstration based on the results obtained.

**Table 5.5 Analytical Locations and Frequency**

| Analyte | Locations          | Start-Up Testing<br>(2 weeks) | System O&M<br>(6 months)                 |
|---------|--------------------|-------------------------------|------------------------------------------|
| COD     | SP1, SP2, SP4, SP7 | Every 1 to 3 days             | Twice per month                          |
| BOD     | SP1, SP2, SP4, SP7 | 2 to 4 times                  | Bi-monthly at SP1 and SP7 only           |
| Anions  | SP2, SP4, SP5, SP7 | 2 to 4 times                  | Twice monthly for 4 months, then monthly |
| Ammonia | SP1, SP7           | Once                          | Bi-monthly                               |

| Analyte         | Locations          | Start-Up Testing<br>(2 weeks) | System O&M<br>(6 months)                 |
|-----------------|--------------------|-------------------------------|------------------------------------------|
| TOC             | SP2, SP4, SP7      | Once                          | Monthly                                  |
| TDS             | SP2, SP4, SP7      | Twice                         | Twice monthly for 4 months, then monthly |
| Turbidity       | SP2, SP7           | Twice                         | Twice monthly for 4 months, then monthly |
| Color           | SP2, SP7           | Twice                         | Twice monthly for 4 months, then monthly |
| pH              | SP2, SP4, SP5, SP7 | Daily                         | Twice monthly for 4 months, then monthly |
| Total Coliforms | SP1, SP7           | Twice                         | Bi-Monthly                               |
| Free Chlorine   | SP4, SP5, SP7      | 2 to 4 times                  | Twice monthly for 4 months, then monthly |
| Perchlorate     | SP2, SP4, SP7      | Twice                         | Twice monthly for 4 months, then monthly |
| HAA5            | SP2, SP4, SP7      | Twice                         | Twice monthly for 4 months, then monthly |
| THMs            | SP2, SP4, SP7      | Twice                         | Twice monthly for 4 months, then monthly |

In addition to data obtain from sampling, data was be collected and recorded from on-line instrumentation shown in Figure 5.5 at intervals of 10 minutes. Voltage and amperage data from the power supply was also be recorded. On-line data was b were viewable in real-time through the cellular data connection to the PLC, and data files was be downloaded from the PLC on a daily basis and added to a database that was be viewable through a Qlik Sense interface.

### ***5.6.2 Monitoring for Energy Demand and Potential Energy Recovery***

Voltage and current to the electrical chemical cell were recorded at the PLC in 10-minute intervals. Gases produced at the cathode were not sampled.

#### **Automated Field Measurements**

The pilot skid featured online measurement of COD, chlorine, and pH (Table 5.4). These instruments recorded values in a data file at the PLC every 10 minutes, and they were used in the treatment control scheme as described in Section 5.3.1. Each day, the data file was appended to the project database, and the data available through a Qlik Sense interface for advanced data visualization.

**Table 5.7 Location of Online Instrumentation**

| Instrument | Measurement Location  |
|------------|-----------------------|
| pH-1       | T-100 (Anodic Tank)   |
| Cl-1       | T-100 (Anodic Tank)   |
| COD-1      | T-100 (Anodic Tank)   |
| pH-2       | T-200 (Cathodic Tank) |
| pH-3       | Finished Water Tank   |
| Cl-2       | Finished Water Tank   |
| COD-2      | Finished Water Tank   |

## 5.7 SAMPLING RESULTS

All data were analyzed to quantify changes between the influent (or, pre-electrochemical treatment) water and that of the effluent water. Averages with 95% confidence intervals were calculated for the aggregate data (influent and effluent) collected over the 5-month operating period. The average (with 95% confidence intervals) fractional removal of the analytes listed in Table 1.1 also was determined, so that the relative treatment effectiveness can be assessed independent of graywater strength (since we expect the graywater strength and composition to vary over time).

Efforts were made to correlate any variable graywater characteristics with final treated water quality. For example, higher salinity graywater may result in elevated levels of free chlorine and/or disinfection by-products.

### 5.7.1 Filtration/COD/BOD

COD/BOD data was used to determine the rate at which the EC system is treating the graywater. Based on the bench-scale testing, the rate of COD removal is expected to be linear. Thus, COD removal rates and the specific charge needed to attain COD removal objectives was determined via linear regression of the data. Changes in the rate of COD removal over time also was assessed.

#### Filtration

The system contained a pre-filter (Filter-1 Figure 5.18) to remove large objects, solids, and hair from the shower water followed by two cartridge filters that removed solids down to 5 microns (Filter-2a and Filter-2b, Figure 5.2). Analytical COD, BOD, and TOC results of the filtered water prior to electrochemical treatment results. Filtered COD, BOD, and TOC analysis over the operation averaged  $113 \pm 27$  mg/L,  $27 \pm 16$ ,  $27 \pm 7$  (Figure 5.19).

#### BOD

After filtration, the greywater entered the electrochemical treatment cell with anode treatment first followed by the cathode treatment. The results show that the BOD was almost completely removed from the anode, cathode, and final effluent for the first 3.5 months of operation and little anionic and final removal for the last 1.4 months of operation. During the first 3.5 months operation the current applied to the electrodes as 12.5 amps while the last 1.3 months the applied

current varied from 6, 4.2 to 2.6 amps indicating some issues within the electrochemical cell. (Figure 5.19 ).

### COD

The untreated COD varied from 131 to 75 mg/L with an average of  $113 \pm 27$  mg/L while the final COD concentrations of the first two sampling events were non-detect (detection limits, 2 mg/L). The COD concentrations for the last four sampling events increased with each event. The percent COD removal initially was greater than 90% and the removal percentage decreased with each following sampling even with a final COD percent removal of 9%.

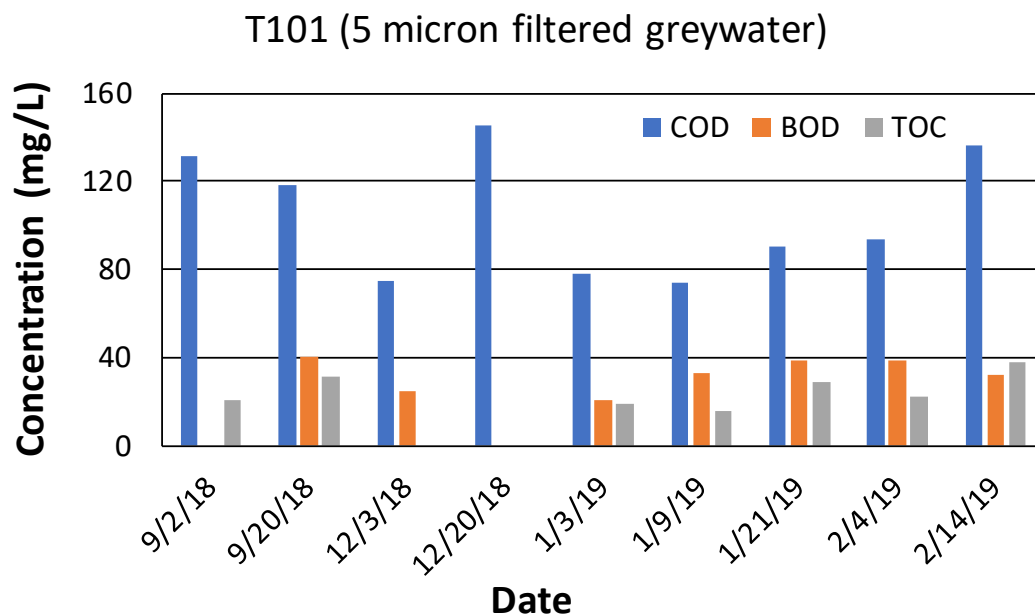


**Figure 5.19. Influent greywater COD, BOD, and TOC results over the demonstration period**

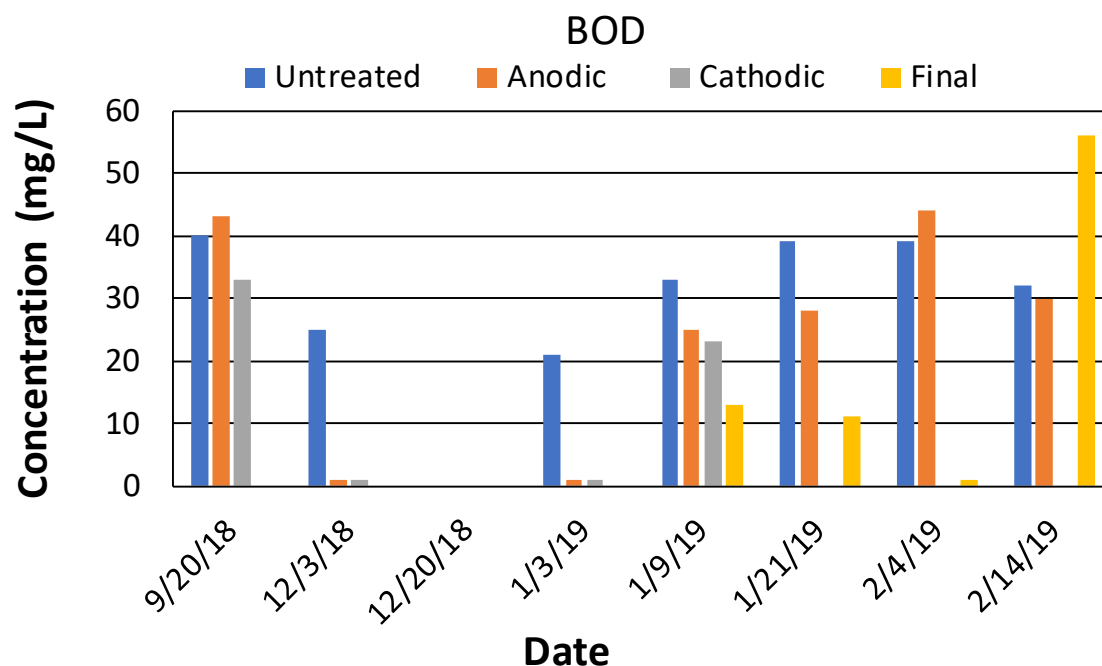


Figure 5.20. BOD of System of duration of demonstration

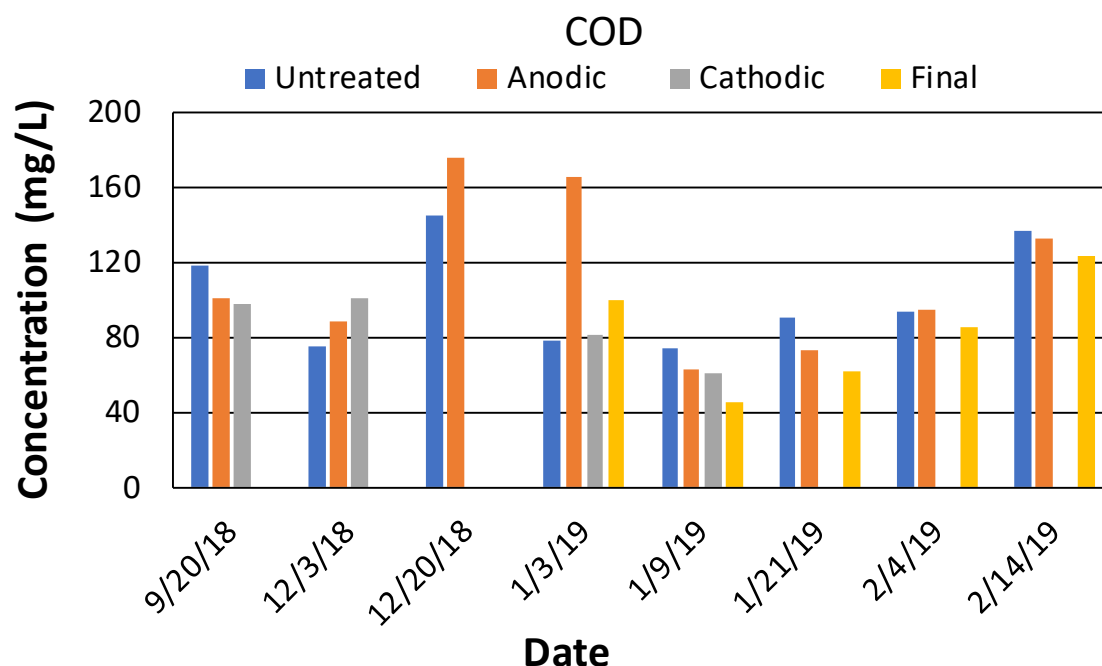


Figure 5.21 COD concentrations of Demo System samples. Where no data exist for the “Final” sampling location, no COD sample was collected. In these instances, sampling at the “Cathodic” location provides a reasonable estimate of total COD removal.

### 5.7.2 Anions, Conductivity, and pH

Water conductivity plays an important role in the amount of current that can be applied to the electrodes. For example, low conductivity was result in a low amount of current that can be applied to the electrochemical reactor. Anion data can be used to estimate the electrical conductivity of the system, which is likely reflected in the applied voltage and overall energy demand needed for treatment. The rate of anodic chloride transformation to free chlorine species, and the subsequent rate of chloride generation from the cathodic reduction of free chlorine species, was quantified. It is expected that the chloride levels in the graywater are proportional to the formation of chlorinated DBPs. The major anions in the influent shower water were chloride and sulfate with a conductivity of 500 to 650  $\mu\text{S}/\text{cm}$  and contained a hardness of 170  $\text{mg}/\text{L}$  as  $\text{CaCO}_3$  and a pH of 7.8. The influent water according to the hardness scale was considered hard bordering on very hard. A sodium sulfate solution of 160  $\text{mg}/\text{L}$  or more was added to the influent water prior to entering the electrochemical cell to increase the conductivity of the influent water.

**Table 5.8 Major anions, harness, and other physical parameters of the influent water.**

| Parameter                                            | concentration |
|------------------------------------------------------|---------------|
| chloride ( $\text{mg}/\text{L}$ )                    | 29 - 66       |
| sulfate ( $\text{mg}/\text{L}$ )                     | 65 - 90       |
| hardness ( $\text{mg}/\text{L}$ as $\text{CaCO}_3$ ) | 170           |
| conductivity ( $\mu\text{S}/\text{cm}$ )             | 500 - 650     |
| pH                                                   | 7.8           |

### 5.7.3 Color, Turbidity, and pH

These data were evaluated with respect to the criteria for potable reuse. These parameters were not tested during the demonstration.

### 5.7.4 Disinfection and Disinfection By-Products

The EC energy needed for COD/BOD removal exceeded that needed for disinfection, pathogen removal was be verified by monitoring for total coliforms. Total coliform levels after EC treatment were be compared to total coliform levels entering the EC cell to determine the log pathogen removal as a function of electrical energy. The initial coliforms were great than 2,420 and decreased to less than 1 (# per 100 mL) during the first week of operation.

DBPs, including free chlorine, perchlorate, HAA5, and THMs, were monitored to with respect to potable water standards. DBP formation was be assessed as a function of the total charge, as well as a function of chloride levels, pH, and time over the 6-month demonstration period. In addition, the cathodic reduction of DBPs was be assessed, and cathodic removal rates were estimated.

Preventing the formation of DBP is difficult. The results show that higher concentrations HAAs were present at higher currents.

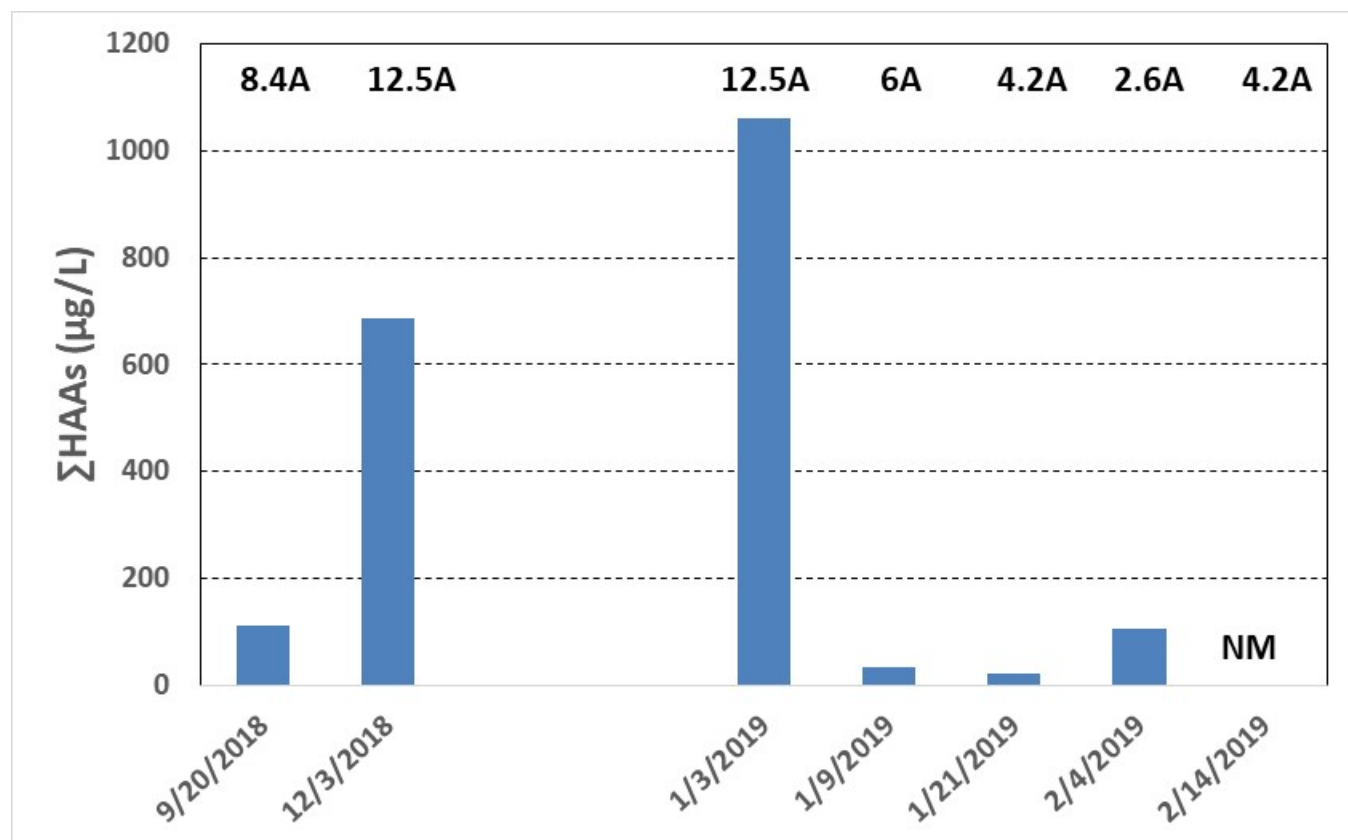


**Figure 22. ΣHAAs concentrations in finished water**

Perchlorate was formed during EC treatment even with the use of MMO anodes. Perchlorate concentrations ranged from 69 to 7000 µg/L with the highest production in the earlier times of operation and the later operational dated the production ranged from 1,000 to over 2,200 µg/L in the finished water.

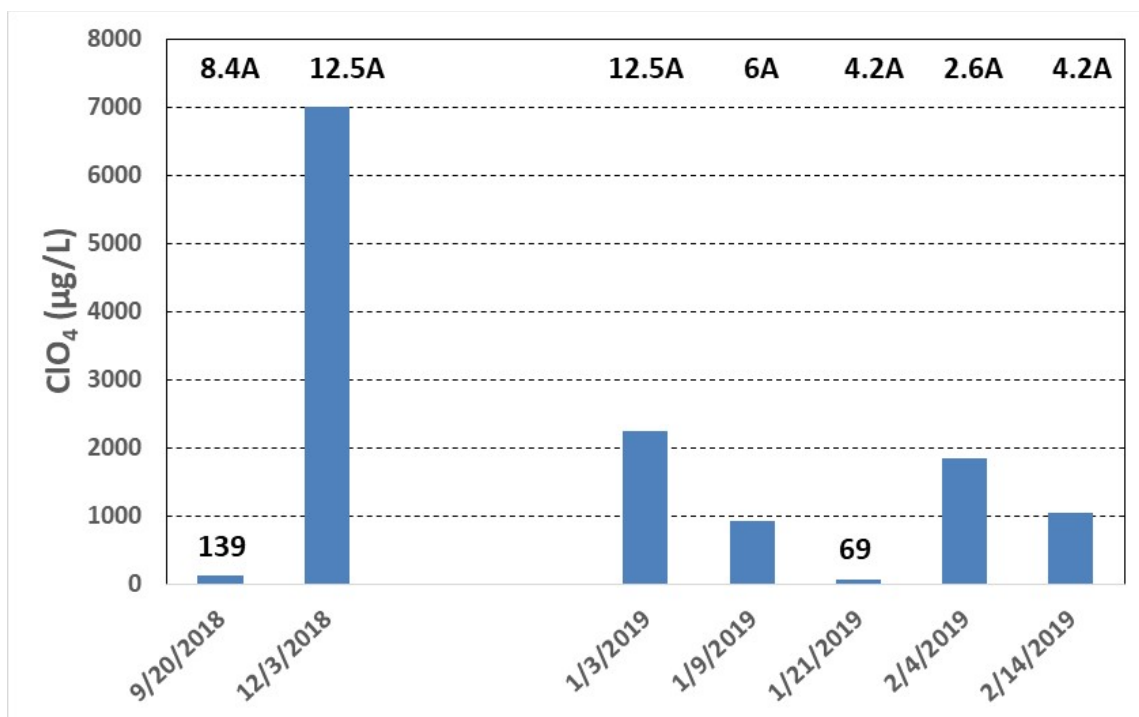


**Figure 23. Perchlorate produce by EC operation**

#### 5.7.5 Overall Assessment – Energy Consumption and Electrode Longevity

Overall performance was based, in large part, on the electrical energy needed for BOD removal. The energy required to remove BOD to less than 10 mg/L range is from 1 to 10 kWh/m<sup>3</sup>, where 4 to 14 kWh/m<sup>3</sup> are required to remove BOD by nanofiltration (NF) membrane bioreactor (MBR). The electrical energy used by electrochemical cell was based on the voltage and current applied to the electrodes over the cell operation. The total EC electricity kilowatt-hours was 793 kWh at a total cost was less than \$100 using the highest rate for calculations. A summary of electrical rates in the Monterey CA by rate type are shown in the table below.

Table Electrochemical electrical total usage and rate cost by type

| Electrochemical Cell energy usage totals |              |            |                      |
|------------------------------------------|--------------|------------|----------------------|
|                                          | Duration (d) | kWh        |                      |
|                                          | 116          | 793        |                      |
| Electrical Energy Cost (\$/kWh)          |              |            |                      |
| Rate                                     | Monterey     | US average | EC cost by rate (\$) |
| residential                              | 0.1559       | 0.1188     | \$ 94.21             |
| commercial                               | 0.1408       | 0.1009     | \$ 80.01             |
| industrial                               | 0.0898       | 0.0667     | \$ 52.89             |

<https://www.electricitylocal.com/states/california/monterey/>

## 6.0 PERFORMANCE ASSESSMENT

This section provides an assessment of the performance of the Dem/Val relative to the performance objectives previously discussed in Section 3. Each subsection discusses the performance relative to an individual performance objective.

**Table 6.1 Demonstration Performance Objectives**

| Performance Objective                                            | Data Requirements                                                                  | Success Criteria                                                                         | Results                                                                             |
|------------------------------------------------------------------|------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
| <b>Quantitative Performance Objectives</b>                       |                                                                                    |                                                                                          |                                                                                     |
| Effective treatment of BOD, COD and total coliforms in graywater | BOD,<br>COD<br>total coliforms                                                     | BOD < 10 mg/L<br>COD <10 mg/L 1 <sup>st</sup><br>Total Coliform <1 (# per 100 mL)        | Achieved<br>Achieve 1 <sup>st</sup> Week only<br>Achieved 1 <sup>st</sup> Week only |
| No issues with disinfection byproducts                           | trihalomethanes<br>haloacetic acids<br>perchlorate                                 | HAA5 <60 ug/L<br>TTHM < 80 ug/L<br>Perchlorate <5 ug/L                                   | Not Achieved<br>Unknown<br>Not Achieved                                             |
| Energy efficiency                                                | Energy,<br>treatment time,<br>determination of net<br>energy hydrogen recovery     | <20 kw-h/m <sup>3</sup><br><br>Hydrogen                                                  | Energy Achieved for BOD only<br><br>Not Achieved                                    |
| <b>Qualitative Performance Objectives</b>                        |                                                                                    |                                                                                          |                                                                                     |
| System longevity and O&M requirements                            | Anode longevity, efforts to control scaling                                        | O&M costs that are less than 80% of current approaches                                   |                                                                                     |
| Scale-up requirements for commercial applications                | Effectiveness of electrode cleaning, COD analyzers, and removal of active chlorine | Mitigation of cathodic fouling,<br>COD analyzer usefulness<br>Removal of active chlorine | Not Achieve<br>Not Achieved<br>Not Achieved                                         |

## 7.0 COST ASSESSMENT/ANAYSIS

A simple cost model was developed to estimate the 20-year lifecycle cost for a full scale electrochemical treatment system for graywater, based on this demonstration. Since many cost elements in this demonstration were unique to electrochemical treatment much of the analysis used was from prior experience in using electrochemical treatment for previous projects.

### 7.1 COST MODEL

The cost model is based on the treatability study, pilot system design, materials, installation materials and labor, start-up, operation & management, training for field operations and removal of pilot system, and site restoration are listed in Table 7.1.

**Table 7.1 Demonstration Project Cost**

| <b>Cost Element</b>                                    | <b>Tracked During the Demonstration</b>                                              | <b>Costs(\$K)</b> |
|--------------------------------------------------------|--------------------------------------------------------------------------------------|-------------------|
| <b>Design</b>                                          | System design and demonstration plan - professional labor                            | \$15              |
| <b>Installation materials</b>                          | includes \$23K for electrochemical cell                                              | \$78              |
| <b>Installation Labor</b>                              | include all the building, programming logic, shipping logisitics, shakedown testing, | \$144             |
| <b>Installation trade labor and materials provided</b> | includes \$10K for electrician and \$10K for plumber                                 | \$20              |
| <b>Start-Up</b>                                        | includes field staff - \$12K and Laboratory analytical costs - \$6K                  | \$32              |
| <b>Operation &amp; Management</b>                      | Field O&M -5. 5 months of operation \$7K per month                                   | \$38.50           |
| <b>Training</b>                                        | \$4K per mid-level person for field training (2 people)                              | \$8               |
| <b>Building Removal and Site Restoration</b>           | includes labor, lifting equipment, lift operator, non-hazardous disposal             | \$12.30           |
| <b>Reporting</b>                                       | Project reporting and meetings                                                       |                   |

#### **Cost Element Bench Treatability Testing**

For this Dem/Val, the team conducted a bench-scale treatability test to determine optimal level of pre-filtration, optimize the EC configuration in terms of COD removal, DBP formation, and energy consumption. The cost of the treatability study was based on the objectives.

**Cost Element –Dem/Val Hardware capital costs**

Off the shelf hair and particulate filtration, pumps, electrochemical treatment cell were used. The greywater control system was custom built with standard components to provide the optimum level of support for the installed treatment system. Expansion to a larger system would require larger filters, pumps, additional electrochemical cells that would be operated in parallel or some variation of serial/parallel depending on site specific BOD and COD requirements. Each additional EC cell would require additional controls that would increase the overall system costs.

**Cost Element- Dem/Val Installation costs**

The installation required shipping the completed greywater treatment system by truck to its site location where it was installed in a 20-foot ISO container. Site contract electricians and plumbers completed the electrical connections in the container and modified the building shower drainage to provide the segregated greywater to the system.

**Cost Element- Dem/Val Operation & Maintenance costs**

The system O&M costs can vary depending on the extent of instrument automation and site conditions and restrictions. Routine O&M tasks were performed by regular field technicians without needing special personnel. The material costs for chemicals and system consumables are project-specific but generally scalable. Professional labor costs were related to initial system start-up operation.

**Cost Element- Hardware lifetime**

The cartridge filters were change once a month as part of preventive maintenance schedule to provide continuous operation. The actual life of the filters and how often they should be changed is unknown. Electrode maintenance and electrode life depend on amount of electric current applied to the electrodes, site specific greywater and potable water hardness. Unscheduled electrode replacement added significant cost to sustainable operations. Other issues included unexpected roof leakage resulting in premature electrochemical cell power supply failure and pH electrode system electronic board failures. The original system was based on the experience of the laboratory treatment that did not include ion exchange columns. Excess water hardness resulted in early failure of the Nafion<sup>TM</sup> membranes between the electrode cells.

## **7.2 COST DRIVERS**

Based on the information and experience obtained from this Demonstration, the main cost drivers to consider when evaluating implementation costs in future projects, including: (1) physical properties of greywater such as pH, hardness, alkalinity, solids (total and dissolved) (2) ability to separate or segregate shower and hand wash basins from black water and laundry water; (3) availability space for treatment system and utilities (electrical and plumbing). These are similar cost drivers for many greywater treatment technologies and not unique to electrochemical implementation. Each of these cost drivers is discussed below. Labor costs were significant for this size demonstration and make difficult to effectively scale to a larger and more long term operation.

### ***Physical Properties of Greywater***

The shower water was potable site drinking water. The greywater properties that altered the drinking water are associated with soap and hair care products used, human hair, some soil or dirt, urine and minor amounts of feces and microorganisms that add BOD and COD to the greywater. The treatment system was designed to remove hair first, followed by remove solids and dissolved solids through cartridge filters. High water hardness must be lowered by ion exchange media to protect the electrochemical system from calcium precipitation and membrane fouling.

### ***Chemical Usage***

Adjustments to pH were made by addition of sulfuric acid to catholyte and sodium hydroxide to the anolyte (Figure 5.2). No other chemicals were required.

### ***Water Reuse***

Applicable water reuse for a larger scaled system would be limited for lawn and fields. The finished water for this demonstration was pump directly into the wastewater system. For larger finished water volumes, a 2,500-gallon underground storage tank was added to the cost. Should the finished water exceed the underground storage available, the excess finished water would be pumped directly into the local wastewater drainage system.

### ***Hydrogen capture***

Because actual electrochemical treatment operations were limited, there is no data available on hydrogen capture for this report. The capture and storage of hydrogen for the electrochemical cell is not included in this analysis.

### ***Utilities***

Availability of space inside building, centralization of plumbing and electrical utilities effect cost. Since an outside building was required, all utilities and storage for the greywater treatment system operation were kept inside the portable system operation building.

### ***Segregation of Shower Water***

In older building and barracks, all the wastewater from showers, hand basins laundry, and toilets are combined. Depending on the physical arrangement of drainage pipes from showers and bathrooms, it may be difficult to separate the shower and hand wash basin water from the black water. Many of the newly constructed multi-story barracks have separate plumbing chases throughout the building for laundry, showers, and laundry for water segregation. If there is no water reuse available, then all finished process water is discharged into the combined wastewater system.

## 7.3 COST ANALYSIS

The cost analysis is based on a 3-floor barracks of the same design as that used in the Dem/Val. remove solids and dissolved solids Demonstration. Each floor will have one shower room including hand basins. An underground storage tank was added to the cost design.

Electrochemical treatment is unique and cannot be easily compared to other types of treatment. Competitive cost analysis with other technologies was not conducted. Comparison of finished water cost will be made but should not be considered reliable.

### 7.3.1 Basis of design

The graywater at the demonstration site, based on the drinking water quality in Monterey County contained a hardness of 170 mg/L as CaCO<sub>3</sub> and a pH of 7.8. The hardness of this water required an ion exchange system to be installed ahead of the filters to remove hardness that resulted in to scaling issues inside the electrochemical cell. Because of this the water quality from the treatability study for this demonstration will be used for the cost analysis. The water hardness for the cost analysis was set to 80 mg/L as CaCO<sub>3</sub> which is considered moderately hard. The lowers the cost of ion exchange media and electrochemical cell maintenance to approximately half of the that in the Demonstration.

- The cost assessment provides a comparison of the EC electrochemical system with an idealized system. The key characteristics of the framework system are as follows:
- The data from implementation at the Dem/Val site and pre-design investigation data are used to support the system design.
- Treatment systems are constructed off-site and transported to target site for installation. Additional electrochemical reactor systems would be constructed in a similar manor.
- The footprint of the treatment system is approximately 8 ft x 20 ft (6.09 m x 2.44 m). The addition of a 3-floor system would require one additional 40-ft ISO storage container.
- Potable water physical properties supplied to the showers and hand basins base characteristics the design including Hardness and pH. Other parameters such as COD, BOD, TSS, and TDS must be sampled from the actual greywater.
- The scale up will be 3-floors requiring on electrochemical cell each with a total flow of 360 gallons per day.
- Each electrochemical cell will treat influent greywater of 90-120 gallons per day over three to 4-hour treatment cycles. Each treatment cycle is based on a 30-gallon batch that is circulated through the treatment system at a rate of 0.1 gallons per minute. Additional electrochemical treatment systems will treat each separate floor using the same volume and treatment rate.
- The site has adequate utilities and there are no concerns for site access or obstructions.
- Treated effluent is non-hazardous and can be discharged to sewer system without additional permitting.

**Table 7.2 Characteristics of raw and filtered graywater from Treatability Study.**

| Analyses                      | Unfiltered | 1 µm Filtered |
|-------------------------------|------------|---------------|
| Chloride (mg/L)               | 33         | 29            |
| Sulfate (mg/L)                | 4.1        | 4.3           |
| Bromide (mg/L)                | <1.0       | < 1.0         |
| Nitrate (mg/L)                | <1.0       | 1.8           |
| Perchlorate (µg/L)            | <3.0       | < 3.0         |
| Chemical Oxygen Demand (mg/L) | 28 / 31    | 20 / 22       |
| Total Organic Carbon (mg/L)   | 5.3        | 4.7           |
| Conductivity (µS/cm)          | 380        | 390           |
| pH (standard units)           | 7.4        | 7             |

### 7.3.2 Cost Analysis

The estimated costs for expanding the pilot electrochemical treatment system are from the cost of the Demonstration operation for 5.5 months. In order to expand the operation from the demonstration to a 1-floor treatment operation treating of 120 gpd scale were used from prior experience because of the uniqueness of this electrochemical treatment system. Similar estimated scale factors were used to expand the treatment to a 3-floor treatment system for a 1-year operation. Individual scale factors for each cost element were calculated with 1-year cost overall scale factor. Similarly, the 3-floor 360 gpd system scale factor was calculated from the annualized cost of the 120 gpd system treatment system. The scale factors from the pilot system to the annualized 120 gpd system was 1.1 and the 360 gpd system was calculated as 2.0. The total annualized cost estimate of the 120 gpd system is \$383,000 and the 360 gpd system was \$764,000 (Table 7.3). The scale up cost from the demonstration to the annualized 120 gpd system captured most of the cost that were built into the high demonstration cost so that the greatest cost increases were labor and additional analytical cost. The annualized cost scaling from a 120 gpd system to a 360 gpd system are additional electrochemical cells and equipment, chemicals, and labor.

An underground finished water storage system was added and provides seven days storage (Table 7.4). If the finished water tank is full, all addition treated water would be pumped to the wastewater drainage system. Another option would be to suspend operation of the EC treatment system until finished water storage become available. Suspension of operation for undetermined lengths of time with full storage are unknown and not considered in the cost analysis (Table 7.5).

The estimate cost of treated water includes all capital and O&M costs (Table 7.6). Using the O&M cost alone do not appreciably change the cost per gallon of treated water. The estimated cost of EC treated water was \$1.25 gal.

**Table 7.3 Estimated cost scaled from pilot operation (5.5 mo.) to annualized cost for 1-floor to 3-floors**

|                                   |                                                           | Pilot        | 1-floor      | 120 gpd      | 3-floors     | 360 gpd      |
|-----------------------------------|-----------------------------------------------------------|--------------|--------------|--------------|--------------|--------------|
| Cost Element                      | Tracked During Demonstration                              | Costs(\$K)   | Scale factor | Costs (\$K)  | Scale factor | Costs (\$K)  |
| <b>Design</b>                     | System design                                             | \$15         | 1            | \$15         | 1.5          | \$23         |
| <b>Installation materials</b>     | electrochemical cell                                      | \$23         | 1            | \$23         | 3.0          | \$69         |
|                                   | Equipment and supplies DC power supply                    | \$20         | 1            | \$20         | 3.0          | \$60         |
|                                   | Instrumentation, programing                               | \$30         | 1            | \$30         | 1.5          | \$45         |
|                                   | shipping                                                  | \$5          | 1            | \$5          | 2.0          | \$10         |
|                                   | chemicals                                                 | \$1          | 1            | \$1          | 3.0          | \$3          |
| <b>Labor</b>                      |                                                           |              |              |              |              |              |
| <b>Installation Labor</b>         | building, programming logic, shipping logisitics, testing | \$144        | 1            | \$144        | 1.5          | \$216        |
| <b>Installation Site labor</b>    | includes electricians and plumbers                        | \$20         | 1            | \$20         | 2.0          | \$40         |
| <b>Start-Up</b>                   | includes field staff                                      | \$32         | 1.25         | \$40         | 3.0          | \$120        |
|                                   | Laboratory analytical costs                               |              |              |              |              |              |
| <b>Operation &amp; Management</b> | Field O&M -5. 5 months of operation                       | \$38.50      | 2            | \$77         | 2.0          | \$154        |
| <b>Training</b>                   | field personnel training (2 people)                       | \$8          | 1            | \$8          | 3.0          | \$24         |
|                                   | <b>Total Annualized Cost</b>                              | <b>\$337</b> |              | <b>\$383</b> |              | <b>\$764</b> |

**Table 7.4 Estimated Cost of Adding Underground Finished Water Storage**

| Cost Element                          | Cost (\$)       |
|---------------------------------------|-----------------|
| 2,500 Gallon Underground Cistern Tank | \$5,100         |
| shipping                              | \$2,500         |
| installation                          | \$7,200         |
| total cost                            | <b>\$14,800</b> |

**Table 7.5 Summary of Estimated Operational Direct Cost Elements**

| <b>O&amp;M costs</b>                                     | <b>Estimated Cost per Year</b> |
|----------------------------------------------------------|--------------------------------|
| Power Supply                                             | 200                            |
| pumps                                                    | 500                            |
| water softening media                                    | 250                            |
| Chemicals                                                | 1,000                          |
| Media Filters                                            | 900                            |
| pH electrodes                                            | 3,000                          |
| 2,500 gal underground storage tank                       | 740                            |
| EC cell BDD cathodes                                     | 2,000                          |
| EC cell Ti/IrO <sub>2</sub> -RuO <sub>2</sub> anodes     | 3,750                          |
| EC Nafion 117 membranes                                  | 800                            |
| <b>Total yearly equipment, supplies, and replacement</b> | <b>13,140</b>                  |
| Installation Labor                                       | 10,800                         |
| O&M Labor Cost                                           | 142,400                        |
| Total Installation and O&M Labor                         | <b>153,200</b>                 |
| <b>Total annualized cost</b>                             | <b>166,340</b>                 |

**Table 7.6 Estimated Cost of Treated Water**

| <b>Cost Element</b>                 | <b>Cost (\$)</b> |
|-------------------------------------|------------------|
| Total System 20-yr cost             | \$3,296,800      |
| 20-yr total greywater treated (gal) | 2,628,000        |
| drinking water cost (\$/gal)        | 0.02             |
| water cost avoided                  | \$52,560         |
| Electrical energy costs             | \$37,580         |
| water treated (\$/gal)              | 1.25             |

### ***7.3.3 Comparison to Other Treatment Systems***

After researching published SERDP and ESTCP project final reports peer review publications for greywater treatment it became evident that the electrochemical greywater treatment was unique and direct comparisons with other technologies. Since the electrochemical greywater system could not maintain continuous operation treatment and cost comparisons with other treatment technologies were not made.

#### ***7.3.4 Cost Estimate Discussion Life Cycle Cost Assessment Summary***

The items with the highest replacement cost are the pH electrodes, EC cell anodes and cathodes (Table 7.5). The pH electrodes are sensitive to high pH and electric field and must be replaced in 6-month intervals. Anode and cathode life may be shortened under continuous operation and by substrate microcracks development in electrode substrates. Cathodes may become coated with oxide precipitate as ion such as  $\text{Ca}^{2+}$  as calcium oxides and carbonates on the surface by electrochemical reduction but in most cases can be cleaned for reuse. Cleaning processes requires removal of the cathodes from the EC cell placed in an acid solution to remove the foreign material. The labor required to perform cathode cleaning may be as expensive as actual replacement.

Chemical costs appear to be high in this analysis as the result of having excess chemicals on-site at the end of the demonstration. For the cost analysis it was assumed that the chemical costs were spread over a full one year period.

The total O&M labor costs were based on the site labor during the demonstration. High labor costs are expected in the demonstration as personnel drive from approximately 100 miles away and stayed in hotels for three nights or more. Labor cost could be lowered over the long term by having local personnel performing maintenance and sampling.

## 8.0 IMPLIMENTATION ISSUES

### *Scaling*

The influent greywater had a hardness of approximately 170 mg/L. This relatively high hardness level facilitated calcium-based scaling in the cathodic compartment of the electrochemical cell. The periodic polarity reversal (up to 10 minutes at the end of every 4 to 6 hour cycle), as well as automated acid rinsing at the end of every cycle, were not able to prevent scale accumulation. Scale accumulation resulted in the applied voltage increasing – ultimately to the point where the desired current density could not be applied. Overall, this resulted in decreased treatment effectiveness and increased energy demands. To address the scaling, water softening (via ion exchange resin) was applied to the water upgradient of the electrochemical cell. This prevented the several scaling issues that were encountered prior to the addition of water softening. Post-demonstration breakdown of the system did reveal some scale accumulation (albeit much less than previously observed), suggesting the need for more aggressive polarity reversal and/or automated acid rinsing than what had been implemented during this demonstration.

### *Humidity impacts*

Given the mild climate in Monterey, CA, climate control was not used in the system shipping container. Unfortunately, this decision resulted in moisture accumulation in the conex box that ultimately damaged the power supply unit after only several weeks of operation. Similarly, the electrochemical cell itself was substantially corroded by the end of the demonstration (although the corrosion of the cell likely did not have any adverse impacts on treatment during the demonstration period). The important lesson learned here is to ensure that proper climate control, particularly humidity levels, are controlled for electrochemical systems.

### *pH electrodes*

While use of the COD meters for this project was experimental in nature (with respect to how useful they would be for this greywater system), the use of pH electrodes for monitoring pH was not intended to be any sort of issue. However, the pH readings were routinely unreliable, which made remote monitoring of system performance problematic. It is unclear as to why the pH electrodes/meter used were problematic. The greywater and electrochemically oxidized water matrix may have substantially shortened the electrode life. It is thus recommended that selected pH measuring instruments be tested for longevity and accuracy at the bench scale prior to field implementation.

### *Perchlorate Generation*

Electrochemically generated perchlorate levels typically ranged between 0.1 and 2 mg/L, which are orders of magnitude above California drinking water levels. While trace (<0.05 mg/L) perchlorate levels were anticipated, the levels generated by the mixed metal oxide anodes in the demonstration system were not anticipated. These results serve as a caution that mixed metal oxide anodes, if used at elevated current density for relatively long time periods, can result in substantial perchlorate formation.

### *Electrode life*

The shortened electrode life may be associated with water hardness and periodic polarity reversal. Couple with electrode longevity was a careful evaluation of the effort needed polarity reversal was used to mitigate scaling at the cathode but was ineffective without the use of the ion exchange media to remove the calcium hardness from the influent water.



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