

Electrochemical treatment for greywater reuse: effects of cell configuration on COD reduction and disinfection byproduct formation and removal

Dina M. Drennan, Raji E. Koshy, David B. Gent and Charles E. Schaefer

ABSTRACT

Electrochemical (EC) treatment presents a low-energy, water-reuse strategy with potential application to decentralized greywater treatment. This study focused on evaluating the impacts of cell configuration, current density, and cathode material on chemical oxygen demand (COD) removal and disinfection byproduct (DBP) formation in greywater. The formation and/or cathodic removal of active chlorine, perchlorate, haloacetic acids, and trihalomethanes were assessed during EC treatment. DBP formation was proportional to current density in undivided EC cells. Sequential anodic-cathodic treatment in divided EC cells resulted in COD removal in the catholyte and anolyte. The anodic COD removal rate (using a mixed metal-oxide anode) was greater than the cathodic removal rate employing boron-doped diamond (BDD) or graphite cathodes, but anodic and cathodic COD removal was similar when a stainless-steel cathode was used. The overall energy demand required for 50% COD removal was 24% less in the divided cells using the graphite or BDD cathodes (13 W-h L^{-1}) compared to undivided cells (20 W-h L^{-1}). Perchlorate formation was observed in undivided experiments ($>50 \mu\text{g/L}$), but not detected in divided experiments. While haloacetic acids (HAAs) and trihalomethanes (THMs) were generated anodically; they were removed on the cathode surface in the divided cell. These results suggest that divided configurations provide potential to mitigate DBPs in water reuse applications.

Key words | anodic-cathodic treatment, cathode materials, decentralized water treatment, disinfection byproducts, divided electrochemical cells, greywater reuse

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ABBREVIATIONS

ANSI	American National Standards Institute	ESTCP	Environmental Security Technology Certification Program
BDCM	Bromodichloromethane	H ₂ O ₂	Hydrogen peroxide
BDD	Boron-doped diamond	HAA	Halo-acetic acid
COD	Chemical oxidation demand	IrO ₂	Iridium(IV) oxide
Cu	Copper	MBRs	Membrane bioreactors
DBCM	Dibromochloromethane	NSF	National Science Foundation
DBP	Disinfection byproducts	O ₂ ⁻	Superoxide radicals
DC	Direct current	O ₃	Ozone
DPD	N,N Diethyl-1,4 Phenylenediamine Sulfate	OH	Hydroxyl radicals
EC	Electrochemical	ROS	Reactive oxygen species
EPA	Environmental Protection Agency		

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RuO ₂	Ruthenium oxide
TBM	Tribromomethane
TCM	Trichloromethane
THM	Trihalomethane
TiO ₂	Titanium dioxide
UV	Ultraviolet
Zn	Zinc

INTRODUCTION

Greywater, defined as wastewater from kitchen, bath, and laundry facilities, represents an important resource for reuse in irrigation, toilet flushing, and similar applications (Ghaitidak & Yadav 2013). Decentralizing water treatment and greywater reuse would alleviate domestic water demands and reduce the volumetric burden to wastewater treatment plants leading to considerable environmental and economic benefit (Imhof & Muhlemann 2005). In the interest of capitalizing on greywater reuse, increased attention has been paid to various decentralization methods such as anaerobic filtering followed by UV treatment (Couto *et al.* 2015), aerobic digestion with hydrogen peroxide treatment for disinfection (Teh *et al.* 2015), TiO₂ photocatalytic treatment for chemical oxygen demand (COD) removal (Chong *et al.* 2015a), and implementation of decentralized membrane bioreactors (MBRs) (Santasmassas *et al.* 2013). Electrochemical (EC) treatment of greywater, while not widely studied, offers advantages over other treatment methods such as operation at ambient temperature and pressures, no waste generation, and its ability to be readily combined with other treatment strategies (Anglada *et al.* 2009; Radjenovic & Sedlak 2015).

EC cells have been studied extensively for their ability to remove organic and inorganic contaminants in various industrial wastewater and water recycling streams (Bagastyo *et al.* 2013a; Radjenovic & Sedlak 2015), as well as for disinfection (Schaefer *et al.* 2017). The treatment mechanisms 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 ($\cdot\text{OH}$), ozone (O₃), superoxide radicals ($\cdot\text{O}_2^-$), active chlorine species, and hydrogen peroxide (H₂O₂) all of which have the

potential to remove COD in wastewater (Gu *et al.* 2006; Chong *et al.* 2015b). Here this concept was similarly applied within the context of greywater treatment.

EC oxidation of chloride-containing water produces chlorine and hypochlorous acid/hypochlorite which reacts with electron-rich moieties to form halogenated disinfection byproducts (DBPs) (Deborde & von Gunten 2008; Prasse *et al.* 2015). Several studies have shown that EC treatment results in formation of trihalomethanes (THMs), and haloacetic acids (HAA), and perchlorate that exceed regulatory levels (Bagastyo *et al.* 2013a, 2013a; Schaefer *et al.* 2017). Thus, mitigation of these DBPs is needed for the effective application of EC treatment for potable water reuse applications.

Several approaches have been studied to mitigate DBPs in electrochemically treated water, including photocatalysis (Uyak *et al.* 2007; Mayer *et al.* 2014), enhanced coagulation, and sorption to powdered activated carbon (Gerrity *et al.* 2009; Mayer *et al.* 2014). Cathodic reduction of THMs has been observed, but with the exception of experiments performed using a Cu/Zn cathode, treatment of trichloromethane has been poor (Li *et al.* 2004; Radjenovic & Sedlak 2015; Wang *et al.* 2016). Other studies have demonstrated the reductive cathodic dehalogenation of HAAs, although complete dehalogenation beyond monochloroacetic acid may be difficult (Korshin & Jensen 2001; Esclapez *et al.* 2012). These findings, coupled with several recent studies that suggest divided EC cell configurations with sequential anodic-cathodic treatment can enhance removal of COD and organic contaminants (Montanaro *et al.* 2008; Yu & Kupferle 2008; Wang *et al.* 2009; El-Ghenymy *et al.* 2014; Schaefer *et al.* 2015).

In this current study, EC treatment for greywater reuse is evaluated. Specifically, we focus on COD removal using both undivided and divided EC cells where the former takes advantage of simultaneous oxidation and reduction whereas the latter separates the anolyte and catholyte with a membrane preventing parasitic consumption of reactive oxidants. In addition, the formation and reduction of DBPs is evaluated along with overall energy demands. Several cathode materials are evaluated. To our knowledge this is the first greywater reuse study to observe the potential for implementing divided EC treatment for COD removal and coupled with cathodic treatment of DBPs.

Table 1 | Characteristics of raw, filtered, and amended greywater

Analyses	Unfiltered	1 μm Filtered	Filtered & Amended
Chloride (mg/L)	33	29	500
Sulfate (mg/L)	4.1	4.3	150
Bromide (mg/L)	<1.0	<1.0	<1.0
Nitrate (mg/L)	<1.0	1.8	<1.0
Perchlorate ($\mu\text{g/L}$)	<3.0	<3.0	<3.0
Chemical oxygen demand (mg/L)	30	21	200
Total organic carbon (mg/L)	5.3	4.7	30.0
Conductivity ($\mu\text{S/cm}$)	380	390	1270
pH (standard units)	7.4	7.1	7.0

All electrochemical experiments were performed using filtered and amended greywater. The greywater was amended after filtering (1 μm).

METHOD

Greywater collection and filtration

Characteristics of the raw and filtered greywater are presented in Table 1. Collection details and amendments to greywater are summarized in the Supplementary Material (available with the online version of this paper).

Electrochemical cell configurations

Parallel plate EC cells (ElectroCell North America, Inc.) were used for all bench-scale testing. The direct current (DC) power source was a Keithley 2220G-30-1 Dual-Output Programmable DC Power Supply. A summary of the

Table 2 | Summary of EC cell configurations evaluated

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

Both undivided and divided cells were evaluated at different current densities and varying electrode materials.

experiments performed is provided in Table 2 and a schematic of the divided cell configuration can be found in Figure 1. Other EC cell configuration information can be found in the Supplementary Material.

Analytical methods are detailed in the Supplementary Material.

RESULTS AND DISCUSSION

Results from the undivided experiments can be found in the Supplementary Material (available with the online version of this paper).

Divided cell experiments

COD removal rates vary between anodic and cathodic treatment

COD removal rates for the sequential anodic-cathodic divided EC cell experiments performed at 12.5 mA/cm^2 are shown for each cathode material in Figure 2. Overall, in the absence of sparging, COD removal was lowest using the stainless steel cathode and greatest using the boron-doped diamond (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 (except for the stainless steel cathode), 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.* 2018b,

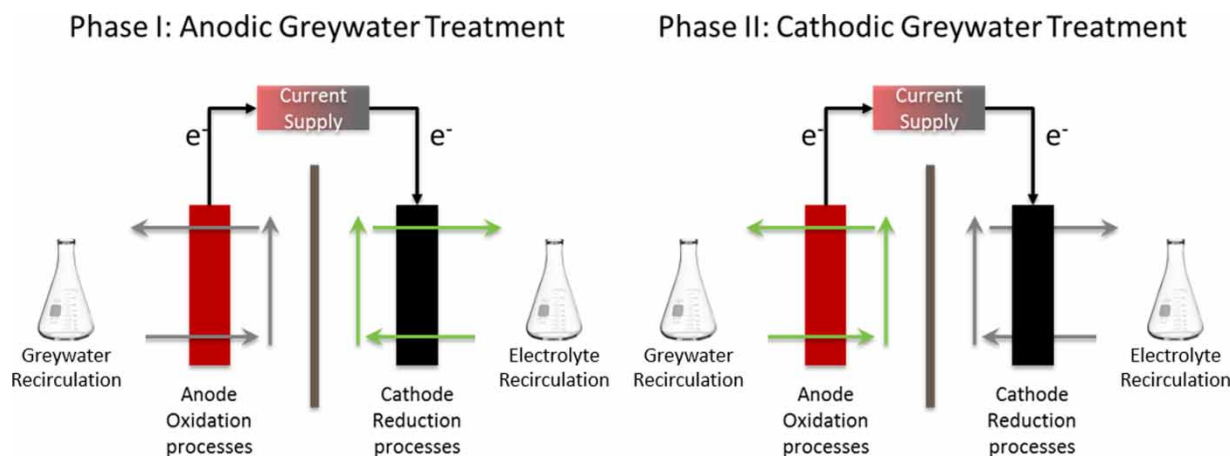


Figure 1 | Schematic of divided cell where Phase I of treatment is anodic and Phase II is cathodic.

2013b). Removal of COD in the catholyte can occur via generation of hydrogen peroxide and/or reduction at the cathode (Rao & 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 2) is not readily explained. Air sparging in the anolyte solution resulted in an increase in the anodic COD removal rate of nearly a factor of 3 (panels (c) and (d) in Figure 2). This increase in COD removal in the anolyte may have been due to enhanced removal of competing electron donors such as Cl_2 . Alternately, sparging could have removed some volatile intermediate oxidation products from the greywater COD.

The net EC 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^{-1} , which is 24% less than that observed in the undivided cell configuration. For the stainless steel (not sparged), the energy demand for 50% COD removal was approximately 20 W-h L^{-1} , as opposed to 17 W-h L^{-1} observed in the undivided configuration. The EC energy demand using a stainless steel cathode with anodic sparging was 11 W-h L^{-1} . These removal efficiencies are comparable to electrocoagulation combined with subsequent electrooxidation with BDD cathodes where a current density of 30 mA/cm^2 resulted in >90% COD removal wastewater at 30 mA/cm^2 (Dermentzis *et al.* 2016).

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 greywater. 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 & 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.

DBP generation and removal in divided cells

Generation of free chlorine and chlorinated byproducts generally differed from observations made in the undivided EC cell experiments. Figure S1 (available online) shows, for all three anodes tested, free chlorine concentrations increased in the unsparged anolyte to between 100 and 200 mg/L, then free chlorine decreased in the catholyte by 45% in BDD, 53% in graphite, and 53% in stainless steel. Free chlorine generation was accompanied by chloride removal during anodic treatment, while molar increases in chloride were observed during cathodic treatment in divided cells as free chlorine was reduced. Free chlorine levels were similar to those observed under

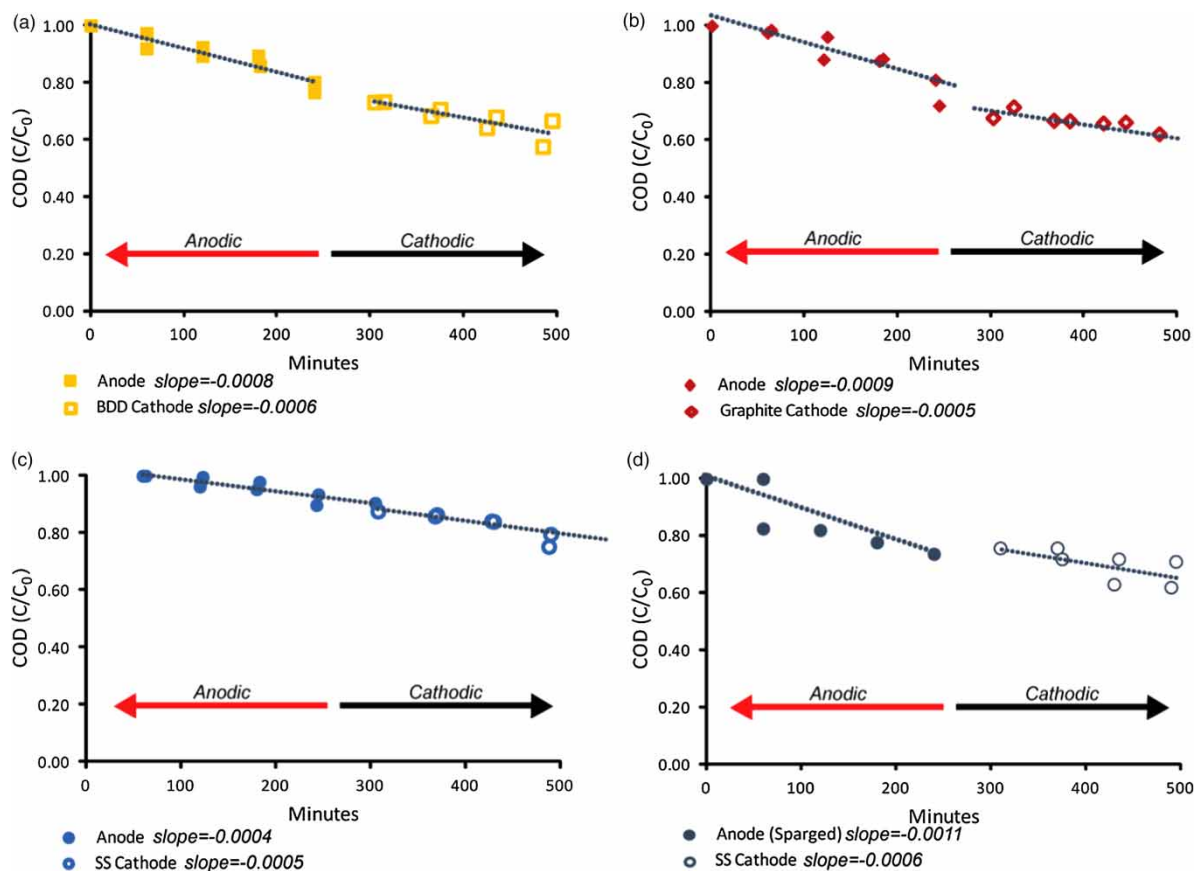


Figure 2 | COD removal in the divided EC cell configuration as a function of time using various cathode materials. All experiments were performed using the Ti/IrO₂-RuO₂ anode, and were performed in duplicate. The applied current density was 12.5 mA/cm², and the cell voltage was approximately 11 V. Cathode materials are specified below each frame and are (a) BDD cathode, (b) graphite cathode, (c) stainless steel cathode, and (d) stainless steel cathode while air sparging in the anolyte solution.

undivided conditions, but the free chlorine in the anolyte (pH = 2.2) was likely present as predominantly Cl₂, 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 was consistent with the higher rate of removal of COD, indicating that a greater rate of oxidation was observed using BDD cathodes (>50% loss in chloride) as opposed to 47% with the stainless steel cathode. In the presence of anodic sparging, free chlorine levels were substantially diminished from approximately 100 mg/L (non-sparged stainless steel cathode experiment) to less than 2 mg/L free chlorine by the end of cathodic treatment due to removal of Cl₂ in the anolyte.

THM results for each of the three cathode materials are shown in Figure S2 and HAA5 results for the BDD cathode are shown in Figure S3 (available online). For each cathode

material, THMs did not exhibit any appreciable accumulation in the anolyte. Previous studies have shown HAA5 formation is favored at low pH (Singer 1994; Liang & Singer 2005; Bagastyo *et al.* 2018b), which likely explains why HAA5 generation was observed, but not THMs, in the anolyte. The rate of HAA5 formation in the anolyte (Figure S3) is similar (within a factor of 2) to that observed for the undivided configuration. Replication of HAA5 data was difficult to achieve especially on the anode side.

Once the anolyte solution was transferred to the catholyte and cathodic treatment was initiated, THM concentrations greatly increased to levels that were 4 times greater than those observed in the 12.5 mA/cm² 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 (Figure S2). The initial increase in THMs upon

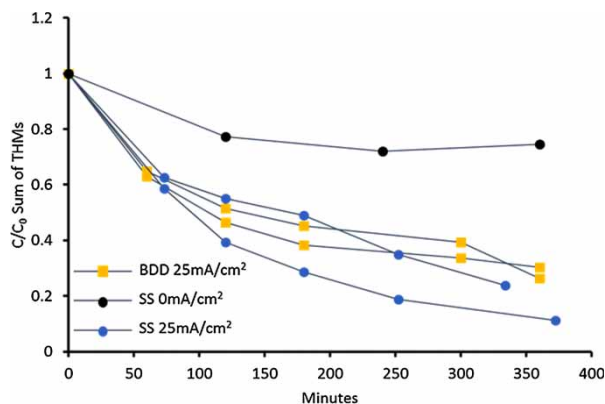


Figure 3 | THM transformation attributable to volatilization (black circles) versus cathodic destruction at 25 mA/cm² using boron-doped diamond (squares) and stainless steel cathodes (blue/grey circles). Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/ws.2018.138>.

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 3), 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 in the anolyte were generally consistent with those observed for the undivided electrochemical experiments (Figure S8, available online). 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 decrease was likely due to cathodic reduction at the anode (Esclapez *et al.* 2013; Garcia-Segura *et al.* 2015). Removal of HAA5 in the catholyte was also observed when using a BDD cathode (Figure S3). Thus, sequential anodic-cathodic treatment provides a potential approach for mitigating the net generation of DBPs.

Perchlorate generation was not observed under divided EC conditions, as perchlorate remained below the analytical

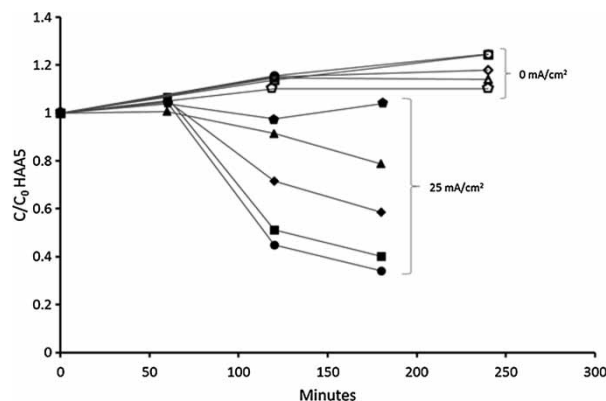


Figure 4 | HAA5 transformation zero current control (open markers) exhibited no and cathodic destruction of individual HAA5 at 25 mA/cm² using boron-doped diamond (closed markers). Circles correspond to DBA, squares TCA, diamonds DCA, triangles MBA, pentagons MCA.

detection limit of 1 µg/L in both the anolyte and catholyte. Thus, operating under divided EC conditions serves as a potential means to mitigate perchlorate formation.

Confirmation of cathodic DBP removal

To further evaluate the cathodic rate of DBP reduction, HAA5 and THM-spiked greywater (not subjected to anodic pre-treatment) was cathodically treated to verify and quantify HAA5 and THM removal. THM results are shown in Figure 3 and HAA5 results are shown in Figure 4. THM losses with zero applied current are likely due to volatilization and/or sorption; no HAA5 losses were observed at zero applied current. The difference in THMs and HAA5 between the control (no applied current) and applied current tests are assumed to be due to cathodic reductive treatment.

Regressed first-order rate constants for the individual THMs are shown in Table 3. Results show THM removal is reasonably described by first-order kinetics. THM removal

Table 3 | Regressed first-order rate constants for cathodic removal of THMs in greywater at 12.5 mA/cm²

Cathode	TCM		BDCM		DBC		TBM	
	Rate Constant (min ⁻¹)	R ²	Rate Constant (min ⁻¹)	R ²	Rate Constant (min ⁻¹)	R ²	Rate Constant (min ⁻¹)	R ²
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

Plus-minus (±) values indicate the 95% confidence intervals. Regressions are normalized to the losses observed in the no current controls. TCM = trichloromethane, BDCM = bromodichloromethane, DBCM = dibromochloromethane, and TBM = tribromomethane.

is generally greater for the stainless steel cathode than for the BDD cathode. For the stainless steel cathode, individual THM removal rates were such that $TCM > BDCM > DBCM \sim TBM$; this trend was not observed for the BDD cathode where all the THMs were removed at the same rate.

HAA5 removal was not reasonably described by first-order kinetics, thus no regression analyses are presented. However, comparison of the zero current controls to the applied current data show that measurable cathodic treatment occurred. Poly-halogenated acetic acids were removed at a greater rate than the mono-halogenated acetic acids, and cathodic reduction of the poly-halogenated acetic acids did not result in any apparent increase in the mono-halogenated acetic acids.

CONCLUSIONS

- Of the configurations evaluated, divided EC cells equipped with BDD cathodes present the most viable option for low energy COD removal in a greywater reuse scenario where COD is removed directly on the anode side and indirectly on the cathode side. Divided cells required less energy to achieve the same COD removal as undivided cells.
- Cathodic treatment using either BDD or stainless steel cathodes resulted in reduction of both THMs and HAA5. Poly-halogenated DBPs were removed more quickly without any accumulation of monohalogenated DBPs.
- Elevated perchlorate concentrations were observed using mixed metal oxide anodes and were not observed using the divided EC cell configuration. To our knowledge this has not been previously observed and should be considered when using MMO anodes for this type of treatment.
- Sparging of an acidic anolyte under divided conditions resulted in decreased DBP formation in the catholyte.
- Collectively, these findings suggest that divided electrochemical cells employing a boron doped diamond cathode and sparging of the anolyte show promise in terms of COD removal and DBP formation management for greywater reuse.

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