Dana Parker July 3rd, 2023 FURSCA 2023

End of Summer FURSCA Report 2023

This summer, my project was on the electrochemical reduction of the carcinogen bromate(BrO_{3}^{-}) in drinking water. The goal of this project was to remove the oxygen atoms from bromate via an electrochemical reduction, with the goal of yielding a bromide ion(Br^{-}) which is not detrimental to human health. This reaction is driven by the use of electrodes which add potential(or energy) to the system.

$BrO_3^-+6e^- \rightarrow Br^-$

This project focuses on finding an electrode that drives this reaction to completion in a timely manner, is sustainable, and cost/resource effective. Within the system, the timing of the reaction, the potential(or energy) applied, and the electrode material can be manipulated. Manipulating these factors allows me to look for reaction parameters that can potentially be scaled up for commercial use, which would effectively remove bromate from our drinking water sources.

I focused on manipulating the working electrode material for my main line of inquiry. To do this, a solution containing bromate is placed in a cell. The electrodes are added to the system, and a bulk electrolysis reaction is run(or potential is applied to the system). After the run, portions of the solution are tested for the presence of bromide. If there is bromide present, we know that bromate was effectively reduced by that specific working electrode. With these results, I investigated which set of parameters would be the most promising in terms of scalability.

I started the summer off by testing the reduction potential of a basic graphite rod. The graphite rod(which is composed of carbon) was used as a working electrode, and it was tested at different potentials found in background literature and at different times. This was done to establish a baseline of carbons reduction potential.

	-2.5 volts of applied potential	tial -2.0 volts of applied potential	
1 hour	Conversion from BrO ₃ - to Br-: 29.6%	Conversion from BrO₃⁻ to Br⁻: 32.6%	
3 hours	Conversion from BrO₃⁻ to Br⁻: 40.4%	Conversion from BrO ₃ - to Br-: 56.4%	
6 hours	Conversion from BrO₃⁻ to Br⁻: Not run	Conversion from BrO₃⁻ to Br⁻: 25.4%	

Table I: Graphite Rod Bulk Electrolysis Data

From these trials, the best conversion was achieved at 3 hours with -2.0V of applied potential.

The ideal parameters established by the graphite rod experimentation were then applied while using bare carbon paper as the working electrode. We started with these parameters as graphite is also carbon, meaning that they are expected to react somewhat similarly.

	-2.5 volts of applied potential:	-2.0 volts of applied potential:	
1 hour	Conversion from BrO ₃ - to Br-: 0%	Conversion from BrO ₃ - to Br-: 0%	
3 hours	Conversion from BrO ₃ ⁻ to Br ⁻ : 0%	Conversion from BrO ₃ ⁻ to Br ⁻ : 0%	
6 hours	Conversion from BrO ₃ ⁻ to Br ⁻ : 0%	Conversion from BrO ₃ - to Br-: 0%	

Table II: Bare Carbon Paper Bulk Electrolysis Data

There was no conversion from bromate to bromide, but this wasn't entirely unexpected as the surface area of the carbon paper was much lower than that of the graphite rod, meaning that the reaction had less room to take place.

After establishing a baseline with bare carbon paper, I moved on to the main part of my project, which was modifying the carbon paper electrode. This was done via the addition of metal-embedded carbon microspheres, specifically palladium-embedded carbon microspheres. This was done by first synthesizing the microspheres. After synthesis, the microspheres were made electrically conductive, turned into an ink, and painted onto the carbon paper. After drying, the modified carbon paper electrode is ready to be used.

The microspheres that I have been testing are 200 ppm palladium-embedded carbon microspheres. I started the modifications with the palladium microspheres as background literature indicated that palladium is the element that is the most effective when it comes to bromate reduction. The results from the bulk electrolysis using the modified working electrode were as follows:

	-2.5 volts of applied potential:	-2.0 volts of applied potential:	-1.0 volts of applied potential:
1 hour	Conversion from	Conversion from	Conversion from
	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:
	0%	0%	0%
3 hours	Conversion from	Conversion from	Conversion from
	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:
	0%	0%	0%
6 hours	Conversion from	Conversion from	Conversion from
	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:	BrO₃⁻ to Br⁻:
	0%	0%	0%

Table III: Modified Pd-CMS Carbon Paper Bulk Electrolysis Data

While there was no complete conversion from bromate to bromide, there was bromite(BrO_2^-) detected, which is an intermediate species between the two. This means that there was a reduction happening; it just did not reduce bromate all of the way to bromide.

After these bulk electrolysis experiments, I pivoted upon my arrival in Ireland. In Ireland, I worked on learning different methods for modifying electrodes. I learned how to drop-cast the

ink directly onto a glassy carbon electrode. Using the drop cast method, I modified the glassy carbon electrode with the ink created with the 200 ppm palladium microspheres that I synthesized in Albion. Using this modified electrode, a bare glassy carbon electrode, and a laboratory sample of 10% platinum on a carbon electrode, I used cyclic voltammetry to measure the effectiveness of each electrode. I did this to establish how the synthesized microspheres will work at a catalyst in an electrochemical system.

I plan to continue using cyclic voltammetry to test the effectiveness of these modified electrodes on the reduction of bromate this fall at Albion College. The data collected from these measurements will allow me to continue to analyze each modified electrode and its potential to be scaled up. I also plan on presenting my current/future results at Elkin Isaac and possibly at the American Chemical Society Conference(s), and base my thesis for the Prentiss M. Brown Honors Program on this work.

This summer, as a whole, has introduced me to the work and experimental design I will experience in graduate school and while I work toward my Ph.D. in chemistry. I would like to thank the Peg 69' and Michael Turner 69' Endowed Scholarship, the Jennifer A. Cook Endowed Fund, and the Richard K. Vitek 56' FURSCA Endowment for supporting my summer research project and related travel. This project has also allowed me to travel abroad for the first time and gain a wealth of knowledge about electrochemistry from many experts, all with different scientific backgrounds. Thank you!