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# Electrochemical-Assisted De-Ionization and Adsoption of Selenium in a Short-Circuit Closed Flow-Electrode Reactor

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# ELECTROCHEMICAL-ASSISTED DE-IONIZATION AND ADSORPTION OF SELENIUM IN A SHORT-CIRCUIT CLOSED FLOW-ELETRODE REACTOR

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Abstract-

The following project was conducted to reduce the concentration of selenium in acid mine drainage. Newly strengthened standards for selenium in fresh water stipulates a challenge within industrial and facilities in the treating of selenium wastewater. Therefore, the development of a promising technology to satisfy maximum limitation of selenium concentration in acid mine drainage is necessary. An electrochemical reactor with activated carbon flow electrodes was applied in this project to validate removal and separation potential of selenium from mining wastewater. In addition, execution of selenium removal were measured at various operative specifications such as, hydraulic retention time (HRT), voltage, recirculation rate, and pH of flow-electrodes for indication of optimum design and engineering parameters. The results indicate nine different sets of data which ran under three different hydraulic retention times of 10, 20, and 30 minutes. With each set of data four reactors were tested each running under a different voltage; zero, two, four, and six volts per cm. Results displayed a decrease in selenium concentration as the HRT and voltage were increased. Furthermore, the conductivity correlated to ion concentration, and as ions were removed the conductivity decreased. Continued experimentation will be used to find the optimal voltage and HRT for Se concentration removal.

## Introduction

Selenium is a natural appearing element that exists in shale, soil, coal, sedimentary rock, and phosphate deposits [1]. There are approximately 40 identified minerals that contain selenium which are infrequent and are normally present along with sulfides of metals such as lead, copper and zinc [1]. Selenium can be discharged into water sources via natural means such as, weathering and by anthropogenic sources, such as exterior mining, irrigated agriculture, and coal-fired power plants [1]. Specifically, high concentrations of selenium have been found in acid mine drainage [2]. This element is ambulatory in surface water and groundwater which makes it capable of being damaging to human health by precipitation and its desorption[3].

Selenium is a worldwide environmental pollutant that largely originates from mining operations, agricultural implementation, coal combustion, and manufacture operations [4]. Contingent on its concentration, when selenium is less than 40 mg/d it can act as a vital micronutrient [5]. Furthermore, if the concentration is above 400 mg/d the element acts as a toxic compound [6]. When found in acid mine drainage the elements concentration fluctuated from 1 µg/L to 7000 µg/L [7]**.** Selenium ranks 147th on the Superfund Priority List of Hazardous Substances of the U.S. Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) [8]. Selenium is listed on the National Primary Drinking Water Regulation table under an inorganic chemical [1]. The standard set for selenium by the United States Environmental Protection Agency (USEPA) for drinking water (2009) is 50 µg/L [9] and for freshwater (2016) is 5 µg/L [10]. Based on these standards especially regarding freshwater it has been challenging for industrial and other relevant facilities to treat their used wastewater [11].

Selenium, can be separated and removed using different methods including adsorption [12,13] , biological reduction [14], zero-valent iron (ZVI) [15,16], electrocoagulation [17], abiotic reduction by green rust [18], photocatalysts [19], electrodialysis (ED) [20], and reverse osmosis (RO) [21]. Consequently, previous research shows that a more stable, simple, efficient, and low-cost techniques are required to remove selenium and to practically apply into industries. Therefore, an electrochemical reactor using activated carbon flow electrodes could deliver a high removal efficiency [22]. Selenium removal could be advanced by a combination of de-ionization, adsorption, and supported by an electrochemical power supply.

Therefore, an electrochemical reactor with activated carbon flow electrode were applied in this project to validate removal and separation potential of selenium from mining wastewater.

### Human health effects

Both selenium excess and deficiency can be found throughout the world [23], however, having a deficiency of selenium is rare in Canada and the United States [24]. Intake across the world is irregular and numerous factors can play a role. For example, how much Se is in the soil especially soil where crops are grown, Se content in fodder, soil pH, Se speciation, organic matter or the ions present that can interact with Se [23]. Selenium can cause major health defects; health conditions such as increased mortality, type 2 diabetes, and increased prostate cancer are associated with both Se excess and deficiency [23].

Skeletal muscle is the major site of selenium storage, accounting for approximately 28% to 46% of the total selenium pool [24]. Early indicators of excess intake are a garlic odor in the breath and a metallic taste in the mouth [24]. The most common quantifiable signs of high selenium is selenosis; hair and nail brittleness or loss [24]. Additional indicators include skin rashes, nausea, diarrhea, mottled teeth, irritability, fatigue, nervous system defects, and lesions of the nervous system and skin [24].

### Aquatic and other effects

Selenium bioaccumulates in the aquatic food chain and prolonged exposure in aquatic invertebrates and fish can result in poor growth, reproductive impairments, and even mortality [1]. Selenium can also opposingly outturn juvenile mortality and growth [1]. According to the EPA, “Selenium is also toxic to water fowl and other birds that consume aquatic organism containing excess levels of selenium.”

## Laws, Rules, and Policies

According to the EPA, “Water quality standards (WQS) are provisions of state, territorial, authorized tribe or federal law approved by EPA that describe the desired condition of a water body and the means by which that condition will be protected or achieved. To protect human health and aquatic life in these waters, states, territories and authorized tribes establish WQS. WQS form a legal basis for controlling pollutants entering the waters of the United States.”

Drinking water standards set in place by the United States Environmental Protection Agency (EPA) are established to protect public health. Public water systems are enforced by the National Primary Drinking and Regulations (NPDWR). In 1975, Selenium was added to EPA’s list of 22 contaminants regulated by NPDWR. Selenium is listed on the National Primary Drinking Water Regulation table under an inorganic chemical. The maximum concentration listed to maintain the safety of the public health is 0.05 (mg/L). In the event of long-term exposure EPA lists possible health effects as, “hair or fingernail loss; numbness in fingers or toes; circulatory problems.”

In 1972 the Federal Water Pollution Control Act was revamped, expanded, and became the “Clean Water Act.” Standards for the quality of surface water are found in the Clean Water Act (CWA), they monitor the emitting of pollutants into surface waters. Under the CWA, EPA established recommended criteria for pollutants in surface waters and developed pollution control programs. The CWA made it required to obtain a permit when emitting any pollutant into accessible water from a point source.

EPA also includes criteria for accepted tribes and states to recognize when establishing their water quality standards specific to “organism only” and “water + organism”. These specific requests regarding human health are under Section 304 (a) of the Clean Water Act. Recommendations from the National Recommended Water Quality Criteria include 4200 micrograms per liter for water that is organism only and 170 micrograms per liter for water that is consumed by humans and organisms. However, the maximum contaminant level from EPA could be more rigorous.

## Why Electrochemical?

Electrochemical reactors with activated carbon flow electrodes could deliver the following; 1) it can advance the capability and rate of adsorption through the voltage [19], 2) it can reduce cumulation of ions in the flow electrode [19], 3) it can improve selenium removal magnitude by merging chemical-physical reactions that could complement each other's constraints [12], and 4) cost can be reduced due to reduced electrolyte replacement [19]. Like above mentioned, possibility of efficient selenium removal under high performance and cost-efficiency could be provided by combination of adsorption and de-ionization supported by electrochemical power supply.

## Reactor Set-up

The reactor contains three chambers separated by semipermeable membranes; one being an anion exchange membrane (AEM) and the other being a cation exchange membrane (CEM) (figure 1). The contaminated influent comes in from the middle chamber. Each side chamber consists of a cathode and anode electrical current. Due to the electric field sodium is drawn to the cathode and selenate is drawn to the anode. Sodium selenate is removed from the middle chamber which reduces the conductivity of the middle chamber. Each chamber contains electrodes that is activated carbon granule and these are recirculated to improve overall performance. The volume of the middle chamber has a total of 500 mL with a working volume of 450 mL. The volume of each flow electrode chamber has a total of

750 mL with a working volume of 650 mL. When filing in the activated carbon of 300 grams to each flow electrode the real working volume ends up being 200 mL.

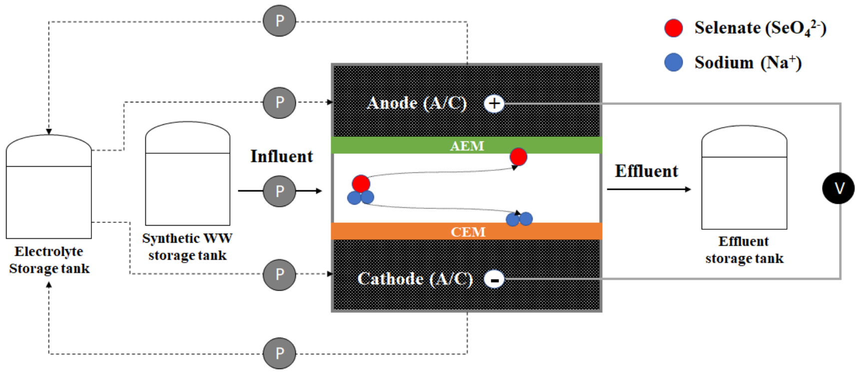


**Figure 1. Reactor schematic**

## Operational specifications

The parameters of the basic set up include: voltage (V/cm), electrolyte pH, synthetic wastewater, electrolyte, and operation type (Table 1). Each reactor is defined by R0, R1, R2, and R3. The reactor titled R0 did not have any voltage being applied, R1 had 2 volts/cm, R2 had 4 volts/cm, and R3 had 6 volts/cm. The electrolyte pH is 6.0 and the operation type is semi-continuous. The solution used for the electrolyte was 5 g/L NaCl and the solution for synthetic wastewater was 10 mg/L $Na_2SeO_4$ with a pH of 6.0.

**Table 1. Basic setup conditions**

| **Parameters** | **Operational conditions** | | | |
|---|---|---|---|---|
| | **R0** | **R1** | **R2** | **R3** |
| **Voltage (V/cm)** | - | 2.0 | 4.0 | 6.0 |
| **Electrolyte pH** | 6.0 | | | |
| **Synthetic wastewater** | $Na_2SeO_4$ (10 mg/L) with pH 6.0 | | | |
| **Electrolyte** | 5 g/L as NaCl | | | |
| **Operation** | Semi-continuous operation | | | |

The hydraulic retention times that applied to the middle chamber of each reactor include: R1 HRT was 10 minutes, R2 HRT was 20 minutes, and R3 HRT was 30 minutes. Retention times were varied by adjusting the influent flow rates (Table 2). For each of the side chambers (FE) where flow electrodes were applied the hydraulic retention times remained the same for each trial in R1, R2, and R3. In reactor R0 no HRT was applied in any chamber as this reactor was used as a control. In the trial where the (FE) chambers had a 2 minutes HRT for R1 the flow rate of the middle chamber was 64.8 L/d, for R2 the middle chamber had a flow rate of 32.4 L/d, and for R3 the flow rate of the middle chamber was 21.6. L/d. The flow rate for the FE chamber for R1, R2, and R3 were all 288 L/d. In the trial where the HRT of the FE chamber was 4 minutes the only change in flow rate was from the FE chamber, this flow rate changed to 144 L/d. Finally, in the trial where the HRT of the FE chamber was 6 minutes the only change in flow rate was from the FE chamber and this flow rate changed to 96 L/d. These conditions were applied in all 9 sets of data which will be discussed later in this paper.

**Table 2. Detailed operational conditions (9 sets)**

| **Hydraulic retention times** | | **Middle chamber** | | |
|---|---|---|---|---|
| | | **10 min** | **20 min** | **30 min** |
| **Flow electrodes (FE) chamber** | **2 min** | Q (middle): 64.8 L/d<br>Q (FE): 288 L/d | Q (middle): 32.4 L/d<br>Q (FE): 288 L/d | Q (middle): 21.6 L/d<br>Q (FE): 288 L/d |
| | **4 min** | Q (middle): 64.8 L/d<br>Q (FE): 144 L/d | Q (middle): 32.4 L/d<br>Q (FE): 144 L/d | Q (middle): 21.6 L/d<br>Q (FE): 144 L/d |
| | **6 min** | Q (middle): 64.8 L/d<br>Q (FE): 96 L/d | Q (middle): 32.4 L/d<br>Q (FE): 96 L/d | Q (middle): 21.6 L/d<br>Q (FE): 96 L/d |

## Measurements methods

For this project the measurement methods include: pH, conductivity, current, selenate and selenite concentrations and A/C surface scanning. The instruments used include: pH probe, conductivity probe, a digital multimeter (DMM), ICP or IC, and a scanning electrochemical microscopy SEM.

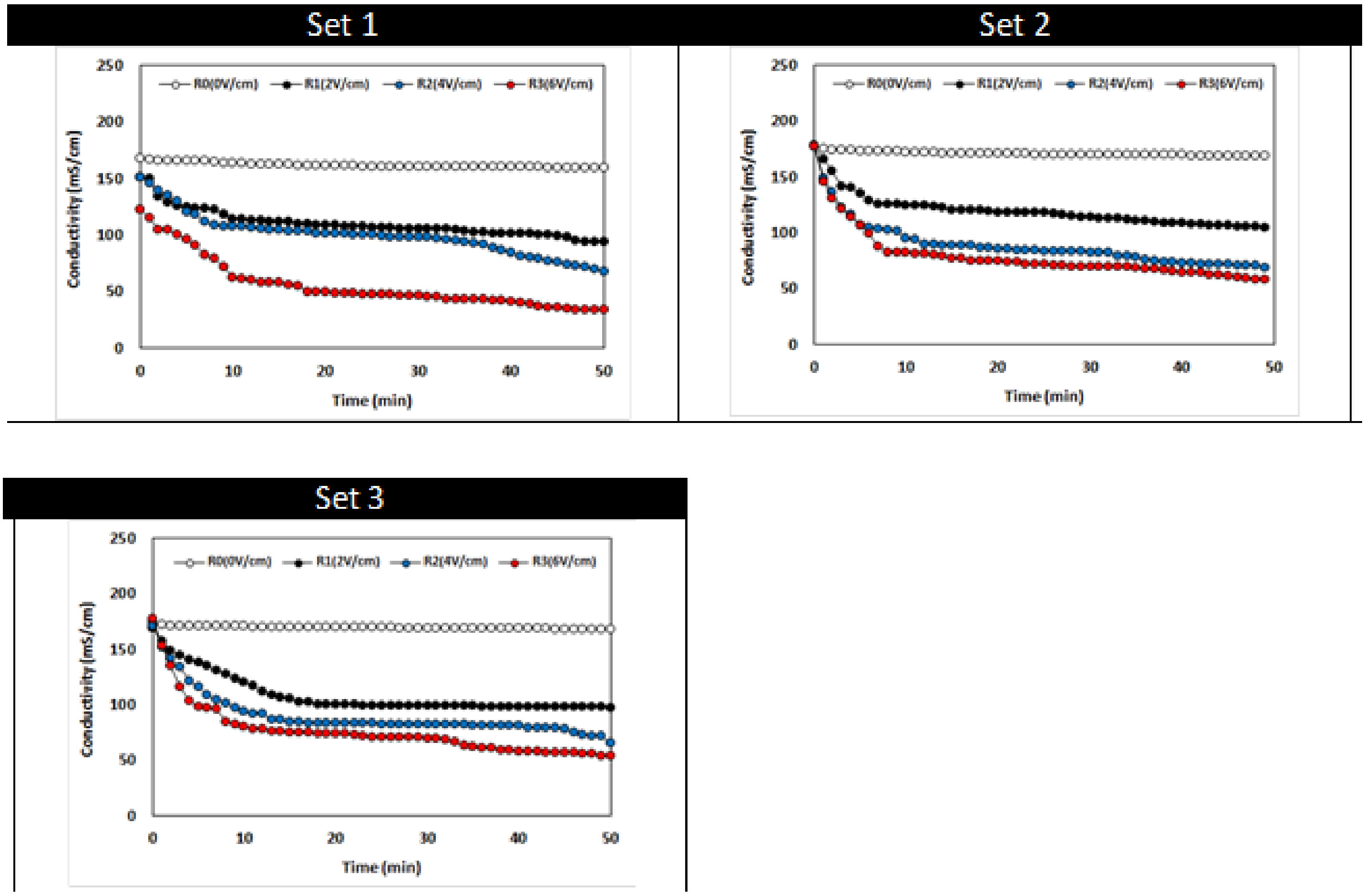
## Data and Results

In the results of set 1-9; the white line represents R0 (0V/cm), the black line R1 (2V/cm), the blue line R3 (4V/cm), and the red line R4 (6V/cm) (figures 2-4). All sets of data ran for 50 consecutive minutes, which is shown on the x-axis. The y-axis shows conductivity in milli siemens per centimeter (mS/cm) at a range of 0 to 250.

In set 1-3 the conductivity of R0 does not change in the 50 minutes (figure 2). In set 1 the conductivity in R1 has the greatest decrease over the first ten minutes and does not drop much more over the remaining 40 minutes. In R2 the conductivity has the biggest decrease in the first and last ten minutes with the final decrease being greater than R1. In R3 the conductivity has the biggest removal in the first 10 minutes but slowly continues to decrease for the final 40 minutes. The conductivity decrease in R3 is almost double the decrease of R1, and R2.

In set 2 the conductivity in R1 has the greatest decrease over the first ten minutes and continues to decrease slowly over the remaining 40 minutes (figure 2). In R2 and R3 the conductivity decrease is also greatest in the first ten minutes and continues to decrease at almost the same rate for the remaining time, however R3 has a slightly more of a decrease. The conductivity decrease in R3 and R2 is almost double the decrease of R1. In set 3 the greatest decrease in conductivity for R1, R2, and R3 lasts for about the first 15 minutes.

The decrease in conductivity in R1 does not change after the first 15 minutes. The R2 and R3 continue to decrease but the change is minimal. In set 1-3 R1 had the least decrease in all sets with an average removal of 38%, R2 had the second for decrease in all three sets with an average removal of 57%, and R3 had the greatest decrease in conductivity in all sets with an average removal of 79%. From this data it is easy to see that the decrease in conductivity has a direct correlation to the amount of voltage being applied.


Set 1

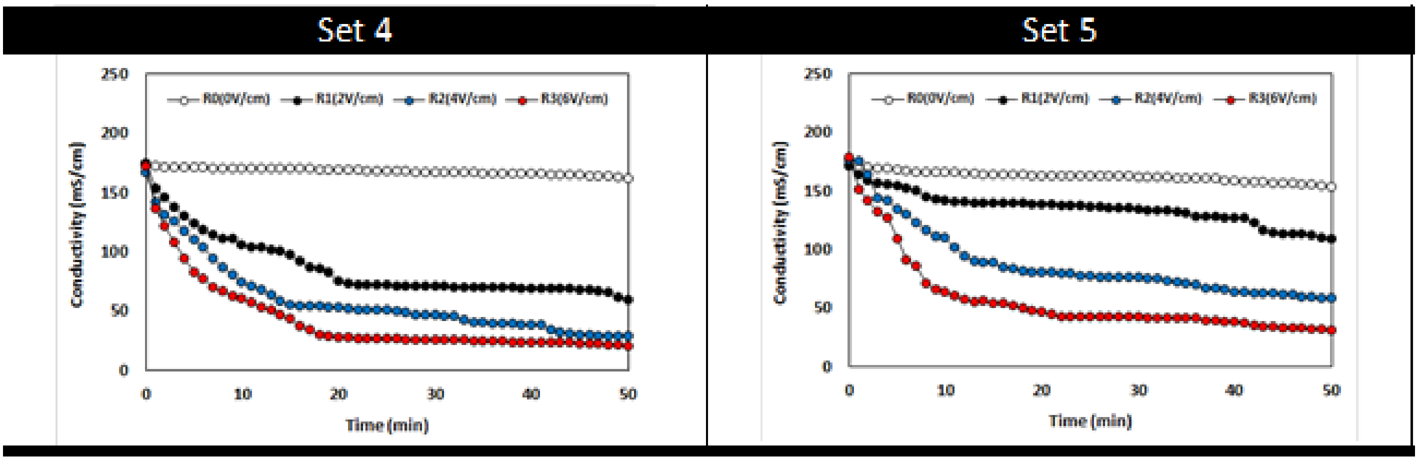
Set 2

Set 3

**Figure 2. Set 1-3: middle chamber HRT 10min**

In set 4-6 the conductivity of R0 decreases very slightly over the 50 minutes due to the increased HRT of 20 minutes (figure 3). In set 4 the conductivity in R1 has the greatest decrease over the first twenty minutes and does not drop much more over the remaining 30 minutes. In R2 the conductivity has the biggest decrease in the first and 15 minutes, with the final decrease being greater than R1. In R3 the conductivity has the biggest removal in the first 20 minutes but slowly continues to decrease for the final 30 minutes. The conductivity decrease in R3 and R2 were both greater than R1.

In set 5 the conductivity in R1 has the greatest decrease over the first ten minutes and continues to decrease slowly over the remaining 40 minutes (figure 3). In R2 and R3 the conductivity decrease is greatest in the first 15 minutes and continues to decrease at almost the same rate for the remaining time, however R3 had more conductivity removal. The conductivity decrease in R3 is almost four times that of R1 and R2 is almost double the decrease of R1. In set 6 the greatest decrease in conductivity for R1, R2, and R3 lasts for about the first 10 minutes and then continues at slow and steady rate. In set 4-6 R1 had the least decrease in all sets with an average removal of 51%, R2 had the second for decrease in all three sets with an average removal of 72%, and R3 had the greatest decrease in conductivity in all sets with an average removal of 83%. From this data it is easy to see that the decrease in conductivity has a direct correlation to the amount of voltage being applied and the longer HRT. In set 4-6 (20 min HRT) the average removal of R1 increased 13%, R2 increased 15%, and R3 increased 4%.



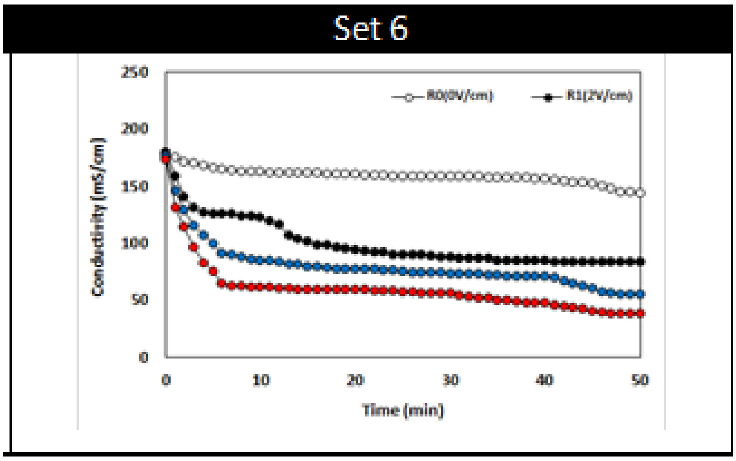
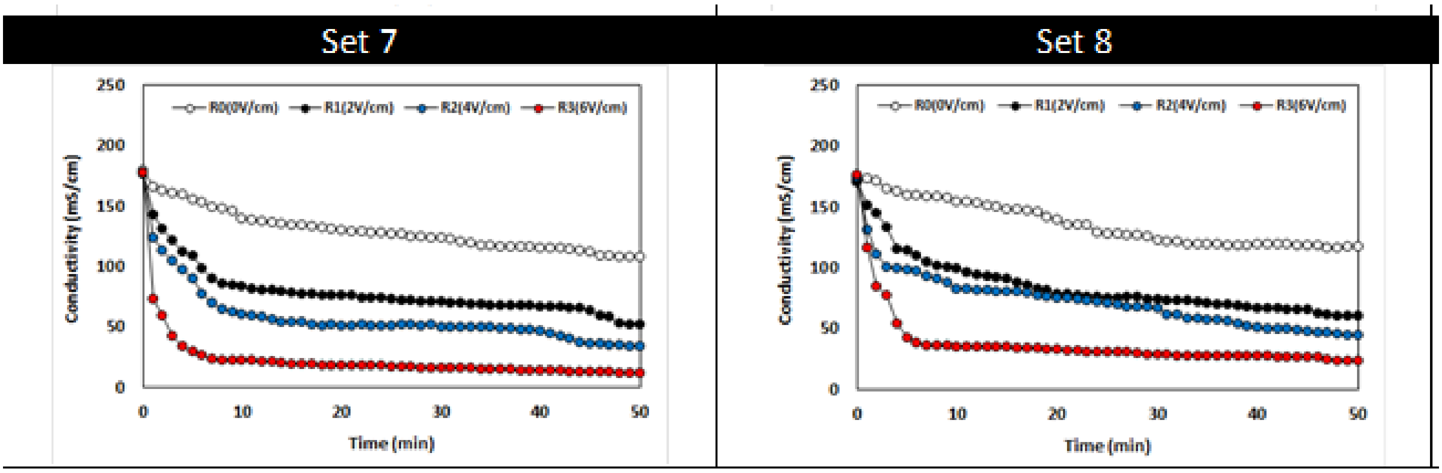


Figure 3. Set 4-6: middle chamber HRT 10min

In set 7-9 the conductivity of R0 is significant enough to record (figure 4). In set 7-9 R0 had the least decrease with an average removal of 30%, R1 had the second for decrease in all three sets with an average removal of 61%, and R2 had a decrease in conductivity in all sets with an average removal of 71%. R3 had a decrease in conductivity from all sets with an average of 84%. From this data it is easy to see that the decrease in conductivity has a direct correlation to the amount of voltage being applied as well as the HRT.



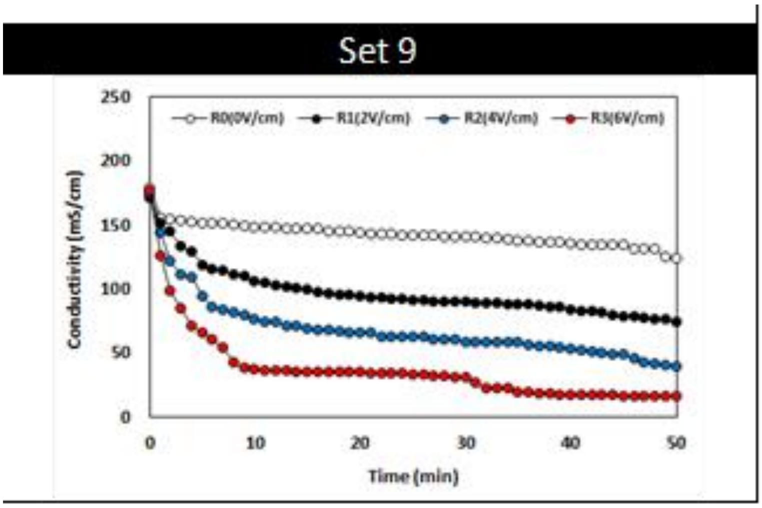


**Figure 4. Set 7-9: middle chamber HRT 30 min**

The conductivity of contaminated water decreases in the reactors over time. The difference between these sets is the hydraulic retention time (HRT) of the middle chamber.

Increasing the HRT improves the removal of Se concentration. The HRT is controlled by the flow rate of the pump, and a higher HRT basically means that the water flows at a slower rate.

**Final conductivity removal**

In the final conductivity removal conductivity reduction efficiency percentage is represented in the y-axis with a range of 0 to 100 (figure 5). HRT of flow electrode per minute is represented in the x-axis with a range of 0-8 minutes. The black color is represented by a HRT of 30 minutes, the red (middle chamber) is HRT 20 minutes, and the blue (middle chamber) is HRT of 10 minutes. It is easily seen in all 3 voltages of 2, 4, and 6 that when the middle chamber has a HRT of 10 minutes the conductivity removed is the least. When the middle chamber has a HRT of 20 minutes the conductivity removed is the second least and when the HRT is 30 minutes the conductivity moved is the greatest. Those results are also increased as the voltage is increased.

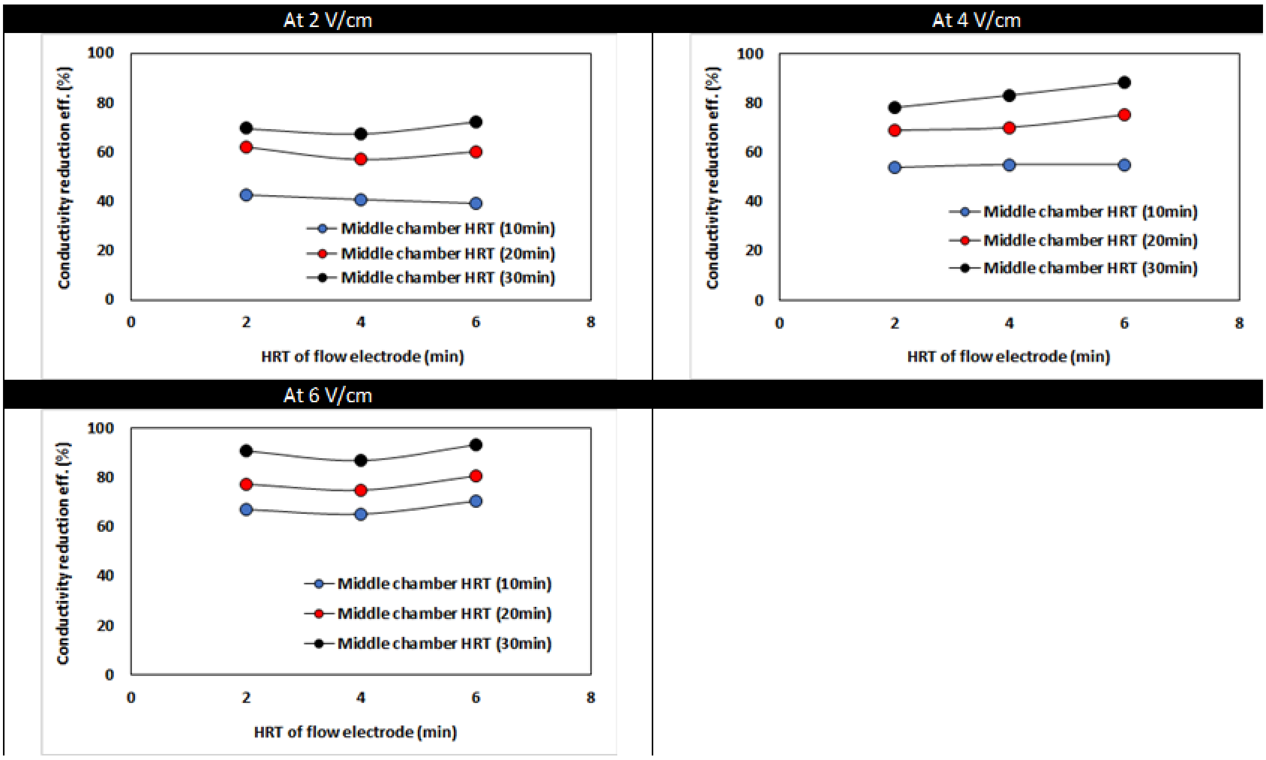


**Figure 5. Data under different voltage**

(i.e., time 50 min in the charts previously) under different conditions

The voltage and the HRT improve the percentage conductivity removal.

## Conclusion

Dependent upon the concentration of Se the element can be beneficial or a harmful toxin. The conductivity is correlated to ion concentration, and as ions are removed the conductivity decreases. As the hydraulic retention time (HRT) is increased the amount of removed Se is also increased. Both the HRT and voltage improve the percentage of conductivity removal. When conducting experiments with an unbalanced concentration between the middle chamber and the electrolyte concerns arose such as ion concentration gradient and an increased resistance. Furthermore, continued testing will be done by some other than myself. Continued experimentation will be used to find the optimal voltage and HRT for Se concentration removal.

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