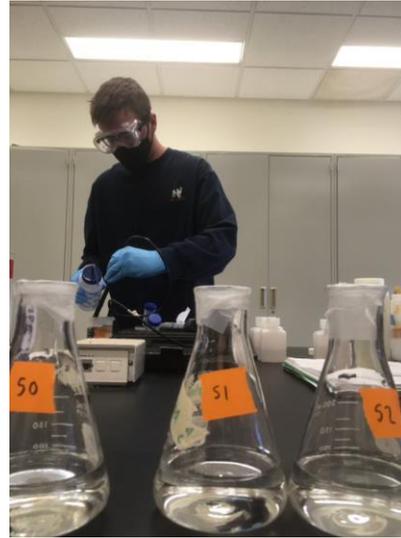


# Using Waste-Based Biochar to Address the Selenium Issue in Western Colorado's Water: Final Report



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### *Executive Summary*

The Colorado and Gunnison Rivers and their tributaries throughout the Lower Gunnison River Basin have dissolved selenium concentrations exceeding the State of Colorado's 4.6  $\mu\text{g/L}$  limit. These exceedingly high concentrations have negative impacts on the organisms, including the four endangered fish species in the Colorado River and its regional tributaries.

Biochar is the carbon-rich substance produced when biomass is heated at relatively low temperatures ( $<700^\circ\text{C}$ ) under low oxygen supply. The study of selenium removal capacities of biochar produced from biomass wastes that are readily available in the Western Slope region remains largely unstudied, and our study aimed to address this research gap.

Our study compared the selenium removal capabilities of two different commercial-grade biochars (purchased from Biochar Now and Wakefield Biochar) over two incubation times (4.5 and 6.0 hours) to determine the impacts of biochar material, iron amendment, and residence time on removal effectiveness. In order to measure the impacts of biochar on selenium removal, samples collected from Leach Creek in Grand Junction, Colorado, were incubated with biochar in batch lab-scale bioreactors. Sulfate was also analyzed to determine its impact on selenium removal effectiveness.

Even though our data showed that one type of biochar—the iron amended variation of the Biochar Now product—was the most effective, none of them were very effective overall. That specific biochar decreased the sample water's selenium concentration (8.09  $\mu\text{g/L}$ ) by 10.1%, but that decrease was only 6.0% greater than the natural reduction that occurred in an untreated sample with no biochar added. Ineffective selenium removal was likely due to high sulfate levels ( $568 \pm 1 \text{ mg/L}$ ) that were over double the legal limit of 250  $\text{mg/L}$ . In fact, concentrations of both selenium and sulfate increased in some of our samples.

Our findings did not necessarily count out biochar as a potential solution to our region's selenium problems, but they did suggest that the two commercial-grade biochars used for this experiment are likely not the most viable options for effectively reducing the selenium content of our local impaired waterways.

## *1.0 Introduction.*

### *1.1 Statement of Regional Problem*

In 2000, the Water Quality Control Commission for the state of Colorado announced a 4.6  $\mu\text{g/L}$  chronic standard concentration for dissolved selenium (*Colorado Water Quality, 2017*). This desired concentration has proven difficult to attain in waterways throughout the Colorado Western Slope, due in large part to the region's underlying geology. The Mancos Shale formation beneath the Grand Valley and the soil derived from it are natural sources of selenium (*Leib, 2008*). According to the research of Butler et al. (1996), basins throughout the Grand Valley featuring extensive outcrops of Mancos Shale also had the highest selenium concentrations. The expansive agriculture of the region also contributes to the problem. Leached irrigation water has been found to react with selenium-yielding Mancos Shale and seleniferous soils to mobilize soluble selenium (*Gates et al., 2009*). Through evapotranspiration, this selenium then concentrates and flows through aquifers into rivers (*Gates et al., 2009*).

The segment of the Colorado River from its confluence with the Gunnison River to the Colorado-Utah border, as well as its many tributaries in the Grand Valley, have been listed on the State of Colorado 303(d) list of impaired water bodies because their dissolved selenium concentrations exceed the 4.6  $\mu\text{g/L}$  standard at the 85<sup>th</sup> percentile level (*Leib, 2008*). Along with the Colorado River, the Gunnison River has also failed to achieve the established concentration

limit. In 2016, 86.5 miles of rivers and streams within the Lower Gunnison Watershed were classified as “impaired” due to selenium concentrations, including the entire 57.6 mile stretch of Gunnison River between its confluence with the Uncompahgre River in Delta, CO and its confluence with the Colorado River in Grand Junction, CO (EPA, 2016).

Elevated levels of selenium in western Colorado’s waterways raise concerns about the health of local aquatic organisms. Selenium is a metalloid that occurs in both inorganic and organic forms and is readily absorbed by organisms, but the organic forms are typically more bioavailable and thus often bioaccumulate to potentially toxic levels (Maier & Knight, 1994). As selenium bioaccumulates through food chains to reach harmful levels, it has been shown to cause reproductive failure, deformities, and other adverse effects in fish, including the threatened and endangered species found in the Grand Valley (Hamilton, 1998). One of the four endangered fish species that the Colorado River and its tributaries are critical habitat for is the razorback sucker (*Xyrauchen texanus*). Research by Hamilton et al. (2004b) found that dissolved selenium concentrations exceeding 4.6 µg/L resulted in rapid mortality of razorback sucker larvae from Grand Valley waterways. Another study also found significant negative correlations between selenium concentrations in adult razorback suckers’ muscle plugs and the percent hatch, egg diameter, and deformities in the embryos of those adults’ eggs (Hamilton et al., 2004a).

In order to better protect the organisms—including the four endangered fish species—that call the Gunnison River, Colorado River, and their tributaries across the Grand Valley home, selenium concentrations must be reduced to achieve the 4.6 µg/L concentration or lower.

## 1.2 Study Objectives

Our study examined biochar as a means to sequester dissolved selenium from contaminated waterways in the Upper Colorado River Basin. Biochar is the carbon-rich substance produced

when biomass is heated at relatively low temperatures (<700°C) under low oxygen supply (Lehmann & Joseph, 2009). We specifically examined the selenium removal capacities of biochar produced from waste biomass readily available in the local region, something that has not been previously investigated. Studying the selenium removal capabilities of biochar from these biomass wastes—in our case, Colorado trees killed by pine beetles and biosolids from wastewater treatment plant—could not only offer evidence supporting a potential selenium remediation technique for our waterways, but also create a beneficial function for otherwise relatively useless organic wastes.

Biochar has been shown to be effective in the removal of unwanted gases such as hydrogen sulfide from anaerobic digesters (Choudhury & Lansing, 2020). Previous studies have also found that biochar can uptake a variety of metal, organic, and inorganic contaminants from soils and water (Ahmad *et al.* 2014), including effective removal of arsenic and molybdenum from ash disposal effluent produced at coal-fired power stations (Johansson *et al.* 2015a).

In terms of selenium removal, Roberts *et al.* (2015) found that biochar could remove 90-98% of selenium from spiked selenium solution in the lab. Johansson *et al.* (2015b) also observed a 98% selenium removal efficiency for iron-amended biochar in a mock solution for selenate ( $\text{SeO}_4^{2-}$ ), the form of selenium commonly encountered in coal mine ash effluent. However, it should be cautioned that removal was low (as low as 3%) and not as effective in the ash effluent and the real-world mine effluent, likely due to the high presence of sulfate (510-640 mg/L  $\text{SO}_4^{2-}$ ) (Johansson *et al.*, 2015a, b). Local water bodies throughout the Western Slope could also have high  $\text{SO}_4^{2-}$  concentrations (concentrations ranging from <10 mg/L to ~1,000 mg/L were observed for the Gunnison River in the North Fork Watershed (Western Slope Conservation Center, 2018)), so it was important that we monitored the sulfate concentrations of the water sampled for this research closely. On the flip side, it is worth noting that reduced selenium removal in the presence

of  $\text{SO}_4^{2-}$  is likely due to sorption competition between the two ions. This means that low selenium removal would most likely be correlated with high sulfate removal, which would still be beneficial for the region's waterways.

Although other studies have found that biochar can serve as a useful removal agent for some gas and water contaminants, there is a lack of research regarding its use for selenium removal from impaired waters in the Western Slope, as well as the use of biochar produced from waste biomass that is available in the Western Slope region. Such biochar is equally important to investigate because these alternative materials likely have varying selenium removal efficiencies due to differences in production (*Clemente et al., 2016*). The use of biochar sourced from local waste biomass would serve as an affordable future selenium remediation method if sufficient research is conducted. Evidence supporting the selenium removal abilities of waste-based commercial biochar could also offer a productive alternative fate for everyday organic wastes produced across the Western Slope. In order to address this research gap, we utilized commercial-grade biochar derived from biosolid waste and local pine beetle kill for the sequestration of dissolved selenium.

Our study thus features three key objectives: 1.) Compare the selenium removal capabilities of biochar sourced from biosolids and biochar sourced from pine beetle kill; 2.) Determine which residence times offer the optimum selenium removal efficiency for each biochar; 3.) Compare the effectiveness of commercially purchased biochar with and without iron amendment. Iron amendment could promote the removal of negatively charged oxyanions by biochar (*Sizmur et al., 2017*). Selenate ( $\text{SeO}_4^{2-}$ ), which is expected to be the dominant form of selenium in the local streams and rivers, is one such oxyanion.

Results from our study could provide specific evidence for the development of technology designed to effectively sequester the high selenium concentrations observed in waterways throughout the Upper Colorado River Basin. Potential future implementation of waste-based biochar in water treatment technologies could also provide a more affordable and beneficial use for a wide range of wastes, such as food waste, municipal solid waste, or even wood clippings of invasive species (Tamarisk and Russian Olives).

## 2.0 Materials and Methods

### 2.1 Sample Water Collection



Figure 1. Collection of sample water from Leach Creek.

Sample water was collected from Leach Creek ~100 meters from its confluence with the Colorado River in Grand Junction, Colorado, in mid-October (*Figure 1*). The sample was stored at 4 °C until use. Prior to the experimental run, the sample was filtered through 0.45  $\mu\text{m}$  filter to remove its suspended solids and was

then stored at 4 °C until use.

Deionized water produced in the Chemistry laboratory at Colorado Mesa University (CMU) was also used in the experiment to determine if the biochar could increase sulfate or selenium concentrations. The deionized water was filtered through 0.45  $\mu\text{m}$  filter and stored at 4 °C until use.

## *2.2 Biochar Source and Amendment*

For our study, two different types of commercial-grade biochar were tested for their Se removal abilities. The first biochar was Wakefield KickStart Biochar (Wakefield BioChar, Columbia, Missouri) and was made of biosolids from sewage waste. The second type of biochar, sold by Biochar Now (size Small—26 mesh to 50 mesh screen size) (Biochar Now, Loveland, Colorado), was produced from trees killed by pine beetles in Roosevelt National Forest in Colorado.

In addition to testing the two commercial-grade biochars as is, these two biochars were also amended with iron in the Environmental Science laboratory at CMU using iron chloride solution. This was done in an effort to determine if iron amendment could improve their effectiveness. Iron amendment was conducted by adding 4.85 g of iron (III) chloride ( $\text{FeCl}_3$ ) to 50 g of each (Wakefield and Biochar Now) biochar. After the biochars and  $\text{FeCl}_3$  were combined, 1 L of deionized water was added and the two respective mixtures were stirred in a Lab-Line 3526 Orbital Incubator-Shaker (Lab-Line Instruments Inc., Melrose Park Illinois) in the CMU Biology laboratory for approximately 48 hours. After stirring was complete, the biochars were filtered through 5  $\mu\text{m}$  filter paper and dried at 105 °C for approximately 48 hours. After drying, the biochars were then rinsed with 1.5 L of deionized water and dried at 105 °C overnight. This method was consistent with that utilized by Choudhury and Lansing (2020), with the difference being that the amounts of biochar,  $\text{FeCl}_3$ , and deionized water were scaled up by a factor of five.

## *2.3 Batch Reactor Testing of Se Removal*

Bioreactor testing was conducted in the Environmental Science laboratory at CMU. Batch lab-scale reactors were used to test the selenium (Se) removal effectiveness of the biochars (2

original commercial-grade and 2 iron amended biochars) over two incubation times (4.5 and 6.0 hours). All treatments were conducted in triplicates. In addition, filtered Leach Creek samples were also incubated without biochar to determine the natural fluctuations in Se concentrations in the water. With all treatments conducted in triplicates, there were a total of 54 reactors, including the Leach Creek samples with no biochar.



Figure 2. Filtration of sample water after biochar treatment.

The reactors (250 mL Erlenmeyer flasks) were filled with 75 mL of their respective water (Leach Creek sample water or deionized water) and 1.25 g of their specific biochar treatments. All reactors were stirred (swirled 10 times) every 30 minutes throughout the duration of their treatment time. After their specified incubation times, the water from each flask was filtered through 0.45  $\mu\text{m}$  filter into two high-density polyethylene (HDPE) plastic storage bottles to be used for Se and  $\text{SO}_4^{2-}$  testing (Figure 2).

#### 2.4 Analytical Methods.

The Se and sulfate ( $\text{SO}_4^{2-}$ ) concentrations in the above-mentioned filtered water samples (Sections 2.1 and 2.3) were measured to determine the concentration before and after biochar treatment. All filtered samples to be tested for  $\text{SO}_4^{2-}$  were stored at 4 °C, while all filtered samples analyzed for Se were acidified drop-by-drop with concentrated nitric acid ( $\text{HNO}_3$ ) until the pH was lower than 2.0. pH measurements were conducted using an EcoSense pH100A (YSI EcoSense

pH100A, Yellow Springs, Ohio). Once acidified, the Se samples were also stored at 4 °C. All  $\text{SO}_4^{2-}$  measurements were conducted within 23 days of treatment, and all Se measurements were obtained within 64 days.

The Se concentrations were quantified using CMU's inductively coupled plasma optical emission spectrometer (ICP-OES) (PerkinElmer Optima 2000 DV, Waltham, Massachusetts). Samples were prepared in accordance with the method specifically developed for use with the ICP-OES (*Bosnak and Davidowski, 2004*) with deviations to the amounts of hydrochloric acid (HCl) and sample water used. Rather than the recommended 20 mL of each, we combined 25 mL of each before boiling. In addition, we used 50 mL glass volumetric flask instead of the suggested 50 mL polypropylene autosampler tubes. Due to the need to repeat some measurements and the limited volume available, some samples were prepared by mixing 5 mL of samples water and 5 mL of HCl for boiling, after which, the samples were transferred to and diluted in a 10 mL glass volumetric flask.

$\text{SO}_4^{2-}$  concentrations were determined using the Turbidimetric Method (*American Public Health Association et al., 2017*) which converted turbidimeter (HACH 2100P, Loveland, Colorado) readings into  $\text{SO}_4^{2-}$  concentrations. Although the method called for 100 mL of each sample and 20 mL of buffer solution, we scaled both variables down by a factor of four, resulting in 25 mL of sample and 5 mL of buffer solution being used for each individual sample. Dilutions of the samples were implemented as necessary if it was found that the turbidity readings exceeded the 10 mg/L  $\text{SO}_4^{2-}$  limit that this specific method was deemed effective for. All dilution adjustments were properly accounted for during final calculations of  $\text{SO}_4^{2-}$  concentrations.

### *3.0 Results and Discussion*

### 3.1 Selenium

Table 3. Average Se concentrations and standard error values for the Leach Creek sample water treatment groups.

Leach Creek Samples: Average Se Concentrations ( $\mu\text{g/L}$ )					
Treatment time (hours)	Wakefield - no Fe	Wakefield - Fe	Biochar Now - no Fe	Biochar Now - Fe	No biochar
0.0	8.09 $\pm$ 0.43				
4.5	8.43 $\pm$ 0.04	8.85 $\pm$ 0.12	7.95 $\pm$ 0.18	7.53 $\pm$ 0.44	7.61 $\pm$ 0.04
6.0	8.72 $\pm$ 0.02	8.12 $\pm$ 0.07	8.49 $\pm$ 0.02	7.27 $\pm$ 0.07	7.73 $\pm$ 0.19

For the most part, Se concentrations in the Leach Creek sample water did not appear to consistently decrease by significant amounts across the residence times or biochars, although the

Biochar Now iron-amended treatments did show Se decreases across both incubation times (Table 3, Figure 4). That specific biochar was the only one to do so. In fact, many of the biochar treatment groups

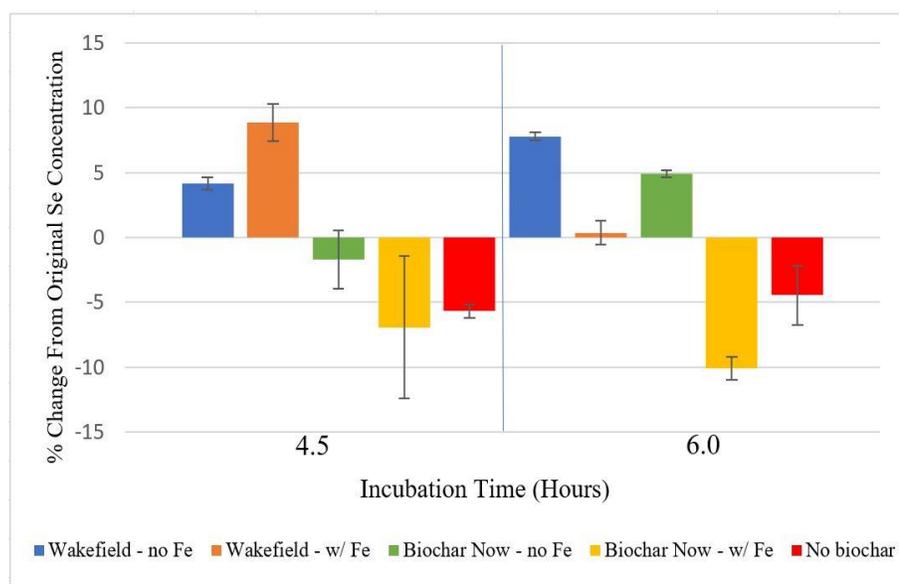


Figure 4. Percent changes in Se concentrations across biochar types and incubation times.

displayed average Se concentrations that were even greater than the average Se concentration of the initial, untreated Leach Creek water (Table 3, Figure 4). For instance, all of the Wakefield (no iron and iron-amended) treatment groups had, on average, 5.4% more Se than the original concentration across both incubation times. Interestingly enough, the average Se concentrations decreased across both incubation times for the samples that were not treated with any sort of biochar. Those untreated samples undergoing 4.5- and 6.0-hour incubation times showed respective decreases of 5.9% and 4.4%.

The biochar/treatment time combination that appeared to most effectively remove dissolved Se was the 6.0-hour variation of the Fe-treated Biochar Now product. It showed a promising 10.1% decrease from the original value, but it is worth noting that its average Se concentration was only about 6.0% less than that of the sample water which was left to sit for the same amount of time without any biochar additions. This finding—as well as the fact that the untreated samples showed greater Se reductions than all of the Wakefield biochar variations—suggests that these commercially-purchased biochars were not very effective Se removal agents.

The results from the deionized water samples appeared to support the underwhelming Se reductions observed in the Leach Creek samples. The deionized water samples and their respective data suggest that the addition of biochar to deionized water may even cause an *increase* in dissolved Se concentrations, as can be seen by the fact that all deionized treatment groups displayed average Se concentrations exceeding that of the original deionized sample (*Table 5*).

*Table 5.* Average Se concentrations and standard error values for the deionized water treatment groups.

Deionized Water Samples: Average Se Concentrations ( $\mu\text{g/L}$ )*				
Treatment time (hours)	Wakefield - no Fe	Wakefield - Fe	Biochar Now - no Fe	Biochar Now - Fe
0.0	$0.37 \pm 0.02$			
4.5	$0.87 \pm 0.12$	$0.56 \pm 0.11$	$0.46 \pm 0.13$	$0.38 \pm 0.18$
6.0	$1.15 \pm 0.02$	$0.81 \pm 0.10$	$0.63 \pm 0.01$	$1.08 \pm 0.45$

\*Low Se values were calculated based on intensities generated by ICP and the calibration curves generated. Values  $< 1 \mu\text{g/L}$  were likely approaching the lower detection limit of the ICP-OES. Lower detection limit testing of the instrument is currently pending.

However, it should be noted that even though the increases can be seen across all of the incubation times and biochars, the Se concentrations in our deionized water samples were already so close to zero to begin with. Despite the minor increases, the Se concentrations in our deionized samples were all still very low.

### 3.2 Sulfate

Table 6. Average  $\text{SO}_4^{2-}$  concentrations and standard error values for the Leach Creek sample water treatment groups.

Leach Creek Water Samples: Average $\text{SO}_4^{2-}$ Concentrations (mg/L)					
Treatment time (hours)	Wakefield - no Fe	Wakefield - Fe	Biochar Now - no Fe	Biochar Now - Fe	No biochar
0.0	568 ± <1				
4.5	492 ± 9	389 ± 1	389 ± 23	321 ± 3	342 ± 37
6.0	483 ± 13	356 ± 22	363 ± 12	429 ± 7	371 ± 5

On average,  $\text{SO}_4^{2-}$  concentrations in all of the Leach Creek samples showed decreases from the original amount (Table 6). These decreases ranged from 13.3% to 43.5% across all biochar types and incubation times, but no clear pattern could be identified among them. For instance, the 4.5-hour variation of the iron-amended Biochar Now product showed a 43.5% reduction in  $\text{SO}_4^{2-}$  content, while the 6.0-hour variation of the same biochar only produced a 24.6% reduction. However, this trend of the 4.5-hour incubation time producing a greater reduction was not consistent, with greater  $\text{SO}_4^{2-}$  reductions in the 6.0-hour treatment observed for three of the five biochar types, including both of the Wakefield biochars.

No clear pattern could be distinguished between iron-amended and non-iron-amended biochars, either. Although the iron-treated variation of the Wakefield biochar produced average reductions that were 20.2% greater those found for its non-amended form across both incubation times, this pattern did not persist for the Biochar Now products, where the average  $\text{SO}_4^{2-}$  reductions for the two types of biochars across both incubation times were nearly identical: 33.8% (no iron amendment) and 34.0% (iron amended).

In addition, the samples that did not receive any form of biochar additions also showed significant decreases in their  $\text{SO}_4^{2-}$  content, including a 39.8% reduction in the 4.5-hour group.

Similar to the Se results, this finding indicates that the observed removal of dissolved pollutants— $\text{SO}_4^{2-}$  and Se—may not be entirely attributed to the biochars that were added.

Table 7. Average  $\text{SO}_4^{2-}$  concentrations and standard error values for the deionized water treatment groups.

Deionized Water Samples: Average $\text{SO}_4^{2-}$ Concentrations (mg/L)				
Treatment time (hours)	Wakefield - no Fe	Wakefield - Fe	Biochar Now - no Fe	Biochar Now - Fe
0.0	14.8 ± 0.1			
4.5	143 ± 36	21.5 ± 0.5	0.06 ± 0.35	1.84 ± 0.37
6.0	156 ± <1	19.5 ± 0.9	0.00 ± 0.52	0.00 ± 2.17

$\text{SO}_4^{2-}$  concentrations in our deionized water samples were relatively uniform for the Biochar Now treatment groups but not for those involving the Wakefield biochar (Table 7). For both of the incubation times for the Wakefield biochar without iron amendment, the deionized water samples saw their  $\text{SO}_4^{2-}$  concentrations increased approximately tenfold from the original reading. The iron-amended version of the Wakefield biochar saw much lower  $\text{SO}_4^{2-}$  concentrations in both of its incubation times than its non-iron-amended version, but its readings were still higher—at least 31.8% greater—than the original deionized water sample. Both the iron-amended and non-iron-amended biochars from Biochar Now reduced the average  $\text{SO}_4^{2-}$  concentrations to zero or nearly zero. Once again, these findings support the trend that the Biochar Now product was more effective than the Wakefield product at removing dissolved pollutants. However, this finding also likely indicates that biochar can actually release dissolved pollutants into water rather than remove them. This possibility is also supported by the handful of Leach Creek and deionized samples that saw their Se concentrations increase after treatment with biochar.

An increased abundance of  $\text{SO}_4^{2-}$  can reduce Se removal (Johansson *et al.*, 2015a, b). With that being said, the very high concentrations observed in our Leach Creek samples—the original sample was more than double the Colorado water quality standard of 250 mg/L—and the notable concentrations in our Wakefield deionized samples likely had some sort of impact on Se removal.

#### 4.0 Conclusion.

Although some of the biochars showed that they may have the potential to decrease dissolved Se concentrations by up to 10%, their use is not likely to be effective in reducing the Se levels of an entire impaired waterway (such as Leach Creek) to the Colorado limit of 4.6  $\mu\text{g/L}$ . Based on our findings, accomplishing such a significant and large-scale decrease would require vast amounts of biochar, whose impacts (besides Se removal) in freshwater river ecosystems are largely unknown. Especially when you compare that ~10% decrease *with* biochar to the ~6% decrease *without* biochar, the results are even more underwhelming. Taking into consideration the fact that many of our Leach Creek samples even showed increases in their Se levels also indicates that their use may not be ideal in natural aquatic ecosystems.

The effectiveness of the biochars in our experiment was minimal, but there were some confounding factors and natural variations that could possibly account for the lack of Se reduction. One such factor that may be inhibiting the Se removal of these biochars is the  $\text{SO}_4^{2-}$  content within the samples. As previously mentioned,  $\text{SO}_4^{2-}$  can inhibit biochar's removal of dissolved Se. Particularly for the Leach Creek samples—where  $\text{SO}_4^{2-}$  concentrations were more than double the legal limit—there was likely significant  $\text{SO}_4^{2-}$  interference preventing the biochars from removing Se at more optimal levels.

Despite the underwhelming removal of Se by the biochars that we observed, further studies should be conducted to more accurately determine their usefulness as removal agents of dissolved pollutants. Further studies could examine biochars produced from different organic wastes than those which our biochars were sourced from. Doing so could help determine the Se removal capacities of varying types of biochars. In addition, different degrees of iron amendment could be studied. Perhaps adding more or less  $\text{FeCl}_3$  solution could enhance the removal capabilities, or perhaps an entirely different sort of amendment could prove effective. Varying incubation times

could also be examined to determine if 4.5 and 6.0 hours are enough time or too much time for a biochar to optimally remove dissolved Se.

### *5.0 Acknowledgements*

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Although our research did not find either of the biochars to be overly effective at removing dissolved Se from a local waterway in the Upper Colorado River Basin, it provided me (Blake Shewmon) with invaluable knowledge and experience. Prior to this opportunity, the extent of my “scientific research” was what I had accumulated through my few Environmental Science laboratory classes during my first two years at CMU. The 250+ hours spent in the field and the lab gave me unmatched experience with a wide variety of instruments, methods, and scientific writing. In a semester when the vast majority of CMU students had limited access to campus and its academic resources, this project allowed me to research an interesting solution to a prevalent local issue with the full arsenal of the CMU Environmental Science Department’s resources at my disposal.

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