

## **Powell River Project 2022-2023 Annual Research Report**

### **Long-Term Effects of Weathering on Mine Soil Properties and Biogeochemical Processes**

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### **Executive Summary**

In the early 1980s, the USDI Office of Surface Mining (OSM) and the Powell River Project (PRP) cooperatively funded the construction and establishment of the Controlled Overburden Placement (COP) experiment. This research project is the longest running of its kind in the world and to date has generated over 50 journal and proceedings articles and numerous student theses and dissertations. With funding from the PRP, we worked to analyze the mid-to-long term (10-30+ years) effects of overburden rock settling and weathering on important mine soil morphological, physical, chemical, and biological properties.

Our main project efforts in 2022/2023 were focused on understanding how mine spoil materials weather and become aggregated over time. We worked to dismantle the mesocosms at the Turfgrass Research Center (TRC) that had previously been filled with a typical SW Virginia sandstone mine spoil (material from the Harlan formation) and allowed to weather over 9.5 years. As part of that process we collected and analyzed intact soil aggregates and clods for their size, bulk density, and elemental composition. We analyzed samples from the following depth increments – 0-20, 20-40, 40-60, and 60-65 cm – from each of the three mesocosms. Elemental composition was quantified using a handheld laser-induced breakdown spectroscopy (LIBS) scanner. We tested homogenized samples of 1) large clods and 2) <2 mm aggregates, and compared them to 3) unaggregated bulk soil, and then analyzed spatial variability of elemental concentrations within aggregates by splitting large clods into surface versus interior subsamples.

In this report, we present results from those analyses focused on the samples collected from the mesocosms. These results complement those presented in our last report (February 2022) that detailed physical, chemical, and morphological changes that occurred during 30+ years of mine soil development. Specifically, the combined results confirm that, while our lab column protocol is a relatively accurate predictor of peak vs. long-term leachate specific conductance (SC; a proxy for TDS), it probably under-predicts peak winter seasonal ion release in younger reactive materials. We continued to see what appears to be a continued gradual decline in TDS elution from the field mesocosm tanks along with a slight seasonal “bump” each winter associated with leaching of accumulated weathering salts from the preceding summer. These results generally support our earlier predictions (e.g., Evans et. al, 2014) of the expected timeframe (15 to 25 years) for field-scale valley fill discharges to fall below current levels of regulatory concern for SC (e.g., 350  $\mu\text{S}/\text{cm}$ ). Combined detailed leaching results from 2020 and 2021 indicate that the

“seasonal bump” may be decreasing in amplitude as these materials continue to weather, but may have actually increased slightly. However, the facts that (a) the deeper spoils in the COP experiment still appeared to be only slightly weathered after > 30 years and (b) the recent apparent small increase in leachate SC from the mesocosms demands that such estimates be made with caution.

Finally, the deconstruction of the Harlan mine spoils in the TRC mesocosms provided the opportunity to gain new insight into aggregation processes. After 9 years of in situ development, the mine spoils had formed many large peds (i.e., > 1-2 cm in diameter) as well as smaller aggregates (i.e., <2 mm in diameter). Larger peds had measured bulk densities that were mostly between 1.6 and 1.9 g cm<sup>-3</sup>, which is in the range of expected values based on other work. The elemental scanning work suggest that metal molecules are more prevalent near the external surfaces of peds, where those elements may help resist disaggregation. Small aggregates collected from the bottom of the mesocosms also had greater resistance to disaggregation, which may imply an incipient cementation process was occurring.

### **Introduction**

The Surface Mining Control and Reclamation Act (SMCRA) of 1977 contained a number of contentious provisions including return to original contour (AOC), long-term liability bonding periods, and return to “equal or better” post-mining land use conditions. However, one of the more important provisions was SMCRA’s allowance for use of pre-selected overburden materials as topsoil substitutes when (1) the native A+E horizon materials are less than 15 cm (6 inches) thick, and (2) the physical and chemical properties of the proposed substitute spoil materials are deemed suitable for such use. Since native topsoil layers throughout the Appalachian coalfields are usually less than 15 cm thick, and removing them from steep slopes is difficult and expensive, the vast majority of coal mined lands in the region have employed topsoil substitutes.

In 1982, the USDI Office of Surface Mining and the Powell River Project (PRP) co-funded the installation of the Controlled Overburden Placement (COP) experiment to objectively assess the viability of the topsoil substitute concept and to determine whether or not organic amendments would be beneficial. One component of the COP experiment directly compared five mixes of sandstone:siltstone (SS:SiS) overburden. All treatments were replicated four times with plots split between herbaceous (dominantly tall fescue) and forest (red oak following pine) vegetation. This experiment was monitored through the mid 2000’s; results can be reviewed at the PRP web site. In summary, this previous work has shown that (1) properly selected and placed spoil materials provided an outstanding soil medium for tall fescue production and allowed vigorous invasion of native herbaceous species; and (2) higher pH spoils – such as the siltstone strata employed – were deleterious to pine tree growth. The COP experiment remains the longest intact and continuously monitored study of mine soil genesis in the world.

Over the past several decades, continued research has shown that relatively unweathered spoils (such as those employed in the COP study) undergo a number of rapid physicochemical changes when placed at or near the land surface. Weathering can release substantial total dissolved solids (TDS) loads to drainage waters over time, which has been implicated as a component of mining related surface water degradation under both low and moderate pH conditions (Clark et al., 2018). Weathering may also affect soil texture, shifting particle sizes towards finer fractions (Lee et al, 2020). Our recent work, which focused on excavating the Turfgrass Research Center (TRC) mesocosms, has shown the formation of clods (i.e., large and dense aggregates typically formed following disturbance) along with smaller aggregates. Subsequent analysis of these aggregates revealed that: (1) large peds had substantial variability in their bulk density; (2) ped exteriors appear to be relatively enriched in cations such as iron and aluminum compared to ped interiors; and (3) micro-aggregates were numerous and most stable near the mesocosms' lower boundary. However, it is still not known if these aggregate characteristics are related to chemical weathering, spoil settling and auto-compaction, or other physical mechanisms such as repeated wetting-drying cycles or gas escape during infiltration.

#### *Progress to Date and Future Research Plans*

During the 2016/2017 project year, we completed the detailed morphological description and basic chemical and physical analysis of soil profiles from selected treatments in the rock mix experiment and surface amendment experiments. Additionally, we initiated a new and innovative column leaching trial to evaluate differences in TDS leaching potentials of the original archived 1982 vs. 2016 rock samples extracted from the plots. The column leaching work was completed in 2018/2019 and the pH and conductance data were reported in our March 2019 annual report.

In 2020/2021 we continued focused analyses of physical and chemical properties of soil samples taken from the detailed pit studies from 2016 vs. original 1982 samples from the same plots (Angel, 2022). Using PRP funding in 2021/2022, we monitored the large mine spoil TDS leaching mesocosms that were placed at the Virginia Tech TRC in early 2013. This effort included routine sampling of leachate and analysis for pH, EC, and ion distributions through summer of 2022.

The mesocosms were emptied in 2022, and the samples collected during that process were analyzed in 2023 for bulk density, aggregate size and stability, and particle size. Preliminary efforts were also conducted to characterize the elemental composition of the exterior versus interior of the larger peds (ranging in volume from 7 to 450 cm<sup>3</sup>) using laser-induced breakdown spectroscopy (LIBS). We also began to develop and evaluate alternative size-separation methods appropriate for the coarse-textured COP plot and mesocosm soils for isolation of light and heavy (mineral-associated) carbon fractions.

Our future research plans are to conclude our analyses on aggregation, elemental composition, and carbon fractions of the soils collected from the TRC mesocosms. We will then perform a similar set of measurements using intact soil samples that we will collect from the COP rock mix experimental plots to better understand how biological versus physicochemical processes lead to auto-compaction, structural development, and carbon storage in these mine soils over time. Finally, we will include the COP plots in a recently funded study to assess soil color as an indicator of organic matter type and content, with the ultimate goal of aiding soil interpretation and management.

### **Objectives**

We have worked to address the following objectives:

1. Quantify the leaching characteristics of fresh mine spoil materials during 9 years of soil development;
2. Quantify and compare the bulk density and aggregate development (e.g., aggregate size distributions) of reconstructed mine soils from the 9-year mesocosm leaching experiment;
3. Analyze concentrations of elemental metals, including iron, aluminum, and manganese, within and between individual peds.

### **Methods**

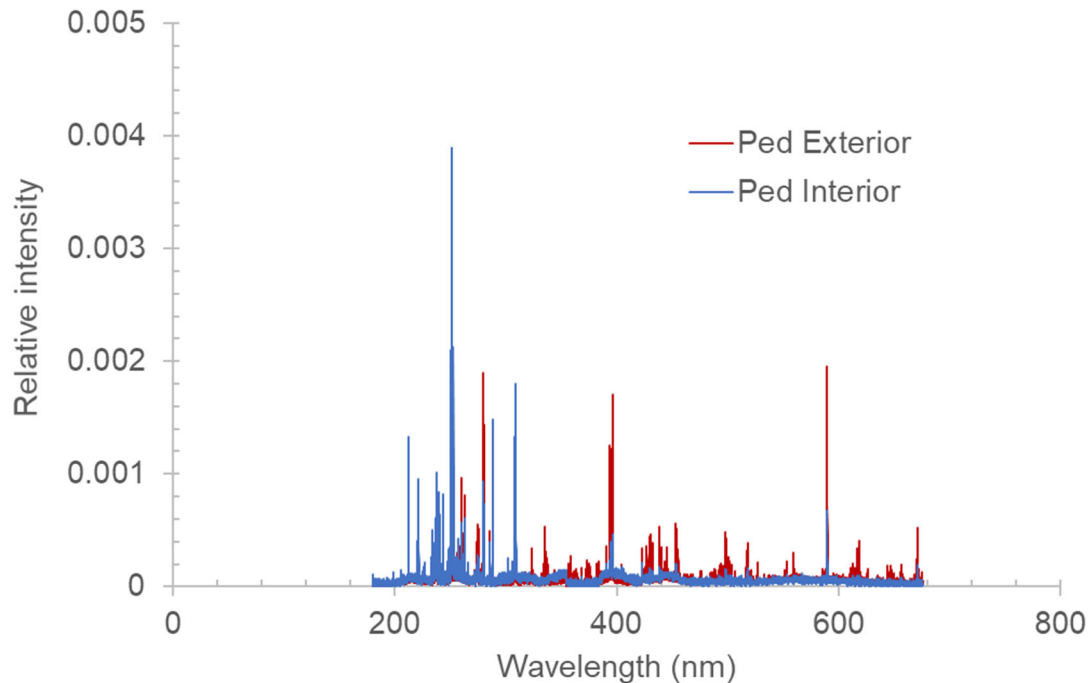
This project is the analysis of a combination of current and past data sets (e.g., mesocosm leaching data), as well as archived and new mine soil samples from the COP experiment that was established in 1982 and field mesocosm experiments established in 2013.

In the summer of 2022, we dismantled the field mesocosms by the following depth increments: 0-20, 20-40, 40-60, and 60-65 cm. For each interval we collected subsamples of the fine fraction, along with any intact peds or clods that were greater than ~2 cm in diameter. We sieved the subsamples to <2 mm and analyzed the sieved material for particle and aggregate size distributions using laser diffraction (CILAS 1140, CILAS Inc., Orleans, France).

We next analyzed larger peds (> 1-2 cm in diameter) for bulk density using a method that we developed with 3D photogrammetry to rapidly measure volume. Two alternative 3D scanning methods were also tested using depth-based infrared and LiDAR sensors; however, those approaches had high variability and low accuracy. Therefore, the results presented in this report only compare the 3D photogrammetry measurements against the traditional paraffin-wax-displacement method for clods (Blake, 1965).

We analyzed representative peds for elemental analysis using a handheld laser-induced breakdown spectroscopy (LIBS) scanner (Z-903, SciAps, Inc.; Woburn, MA). We specifically tested homogenized samples of 1) large clods, 2) <2 mm aggregates, versus 3) unaggregated bulk soil. We then analyzed spatial variability in elemental concentrations within aggregates by splitting large peds into surface versus interior subsamples. Clod volumes were first determined with the previously developed photogrammetry method and the exterior surface was scanned with the LIBS device in three locations. Clods were then split to expose interior surfaces and photogrammetrically scanned to determine the volumes of the split clod pieces. These interior surfaces were scanned with the LIBS device in three locations. The LIBS scanner provided spectral results (example shown in **Fig. 1**) that were converted to relative intensities. Upcoming work will take elemental data from 30 representative samples to convert relative intensities to elemental composition.

Finally, we evaluated possible carbon fraction separation methods to determine the optimum method for observing changes to light (particulate) and dense (mineral-associated) carbon forms (Lavalee et al., 2019) in the coarse-textured COP plot and mesocosm samples. We began an experiment to validate a wet-sieving (to 53  $\mu\text{m}$ ) size separation across standard soil samples with wide variation in soil texture and carbon fraction distribution. This experiment is ongoing, and the validated method will be used for continued carbon fraction characterization of COP plot and mesocosm samples early in 2024.



**Figure 1.** Example of spectra that come from using laser-induced breakdown spectroscopy (LIBS) on the exterior (red) versus interior (blue) of a ped collected from the TRC mesocosms. The x-axis indicates the wavelength (nm) and the y-axis indicates intensity of the spectra.

## **Results and Discussion**

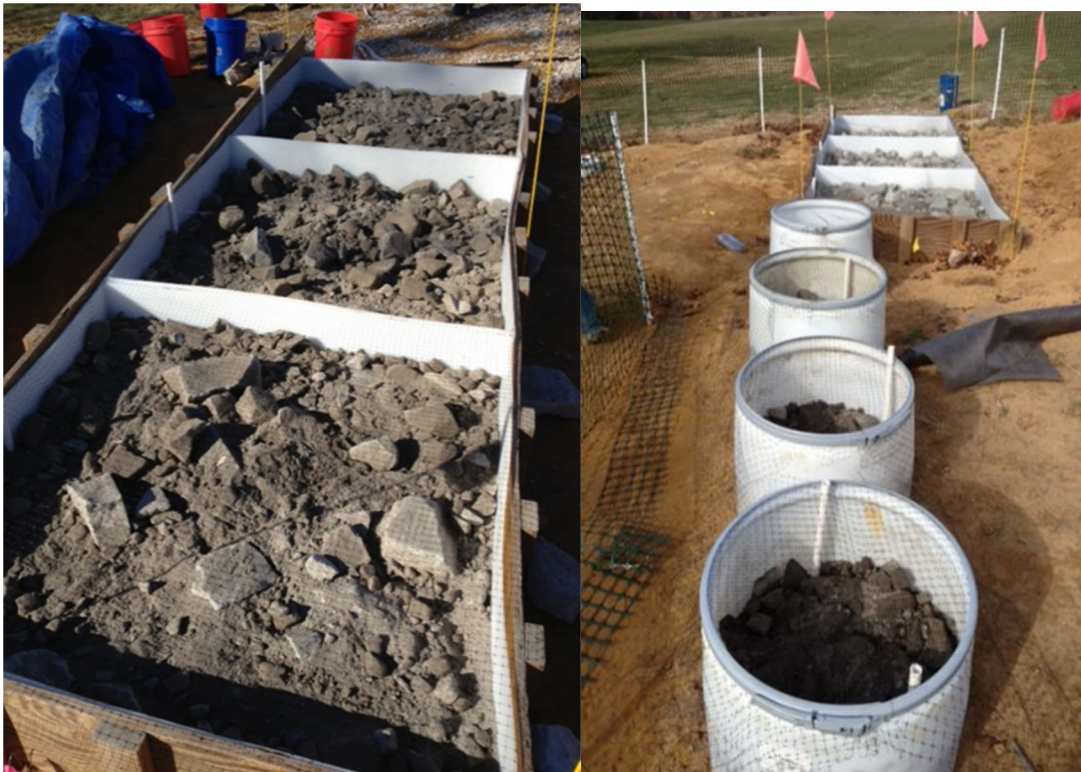
### *TDS Release Over Time by a SW Virginia Sandstone Spoil – Mesocosm Results*

In December of 2012, we initiated a combined lab column and field mesocosm study (Ross, 2015) focused on determining the multi-year total dissolved solids (TDS) elution behavior of a typical SW Virginia sandstone mine spoil (Harlan Fm.) in comparison to our column leaching method prediction method (Orndorff et al., 2015; Daniels et al., 2016; Clark et al., 2018). To accomplish this, we constructed large “leaching tanks” (mesocosms; see Fig. 2) at the Virginia Tech Turfgrass Research Center (VTRC) in Blacksburg and filled them bulk fresh mine spoil collected from the Harlan Formation at the Red River Coal active mine above the Powell River Project field site. This stratum is similar to the un-weathered SS component originally utilized for the COP experiment, but occurs higher in the stratigraphic column. The bulk spoils were loaded by hand into the mesocosms and barrels (**Fig. 2**) to assess the relative influence of experimental unit volume on TDS leaching behavior vs. original column predictions (as described in our last Annual Report in February 2022). Materials placed into the barrels were generally < 15 cm in diameter, but all materials up to ~50 cm in diameter were loaded into the mesocosms. Leachates were drained from below the mesocosms into separate belowground receiving containers and sampled monthly during the winter leaching season and/or following major precipitation events during the growing season. Local rainfall was also collected in an adjacent clean barrel for analysis. All samples were analyzed for pH and SC in our labs within several hours of field collection. The final sampling of leachates produced by the mesocosms occurred in May 2022.

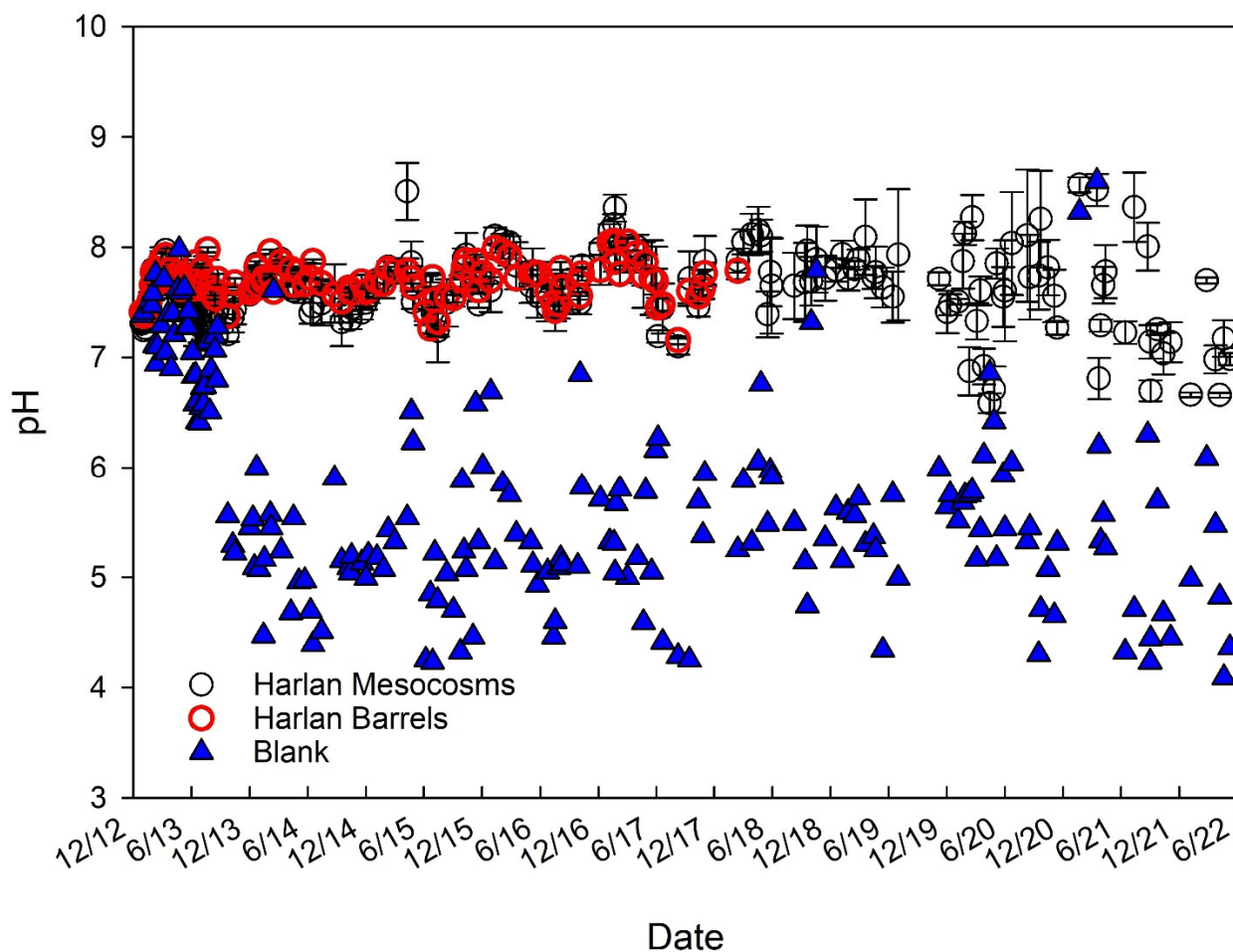
While not covered in detail in this report, the same spoil was also leached under unsaturated conditions in the laboratory (Ross, 2015), based on a method developed by Orndorff et al. (2015) with 7.5 cm (d) x 40 cm (l) PVC columns filled with spoil fragments  $\leq 1.5$  cm. Full details on the effects of saturation, dosing cycle, and leachate pH on eluted pH/SC and metal/oxyanion release are found in Parker (2013). Our previous Annual Reports compared the lab column data vs. the field mesocosms; this report focuses only on the final cumulative field leaching pH and SC response over the full ten-year monitoring period.

In general, the collective leachate pH data (through May 2022) observed in the field was similar between the two sizes of container (i.e., barrels and larger mesocosms (**Fig. 3**). The field data were also well-correlated with the initial lab column results (Ross, 2015; not shown). Similar to many SW Virginia unweathered spoils, these materials are high in pH, most likely due to trace carbonates and other reactive alkaline weathering components like feldspars and metamorphic lithic fragments (Clark et al., 2018). The pH vs. time behavior for the mesocosms (**Fig. 3**) through early winter 2021 reveals a continuing strong seasonal effect, with pH (and presumably total alkalinity) increasing in the cooler winter months through 2019, most likely due to the slight but positive effect of decreasing temperature on pH, CO<sub>2</sub> partial pressures, and the associated carbonate/bicarbonate equilibrium.

The drop in pH over the first two years of the local rainfall (labeled as “blank” in figures) is notable and more than likely indicates that some neutralization must have occurred in the rainwater collection system due to an unknown interaction with the plastic barrels or gravel/sand used in those systems. However, several higher pH rainfall events were noted in later years, particularly 2021. The longer-term rainfall pH ranged from 4.5 to 6.5 as expected. The pH data from 2020 to 2022 also appear to indicate a significant decrease in pH over the summer months indicating that the inherent neutralizing capacity of the spoil may be finally depleted.



**Figure 2.** Harlan spoil leaching mesocosms (left) and barrels (right foreground) at Virginia Tech Turfgrass Research Center (VTRC) in November 2012. Leachates were collected monthly or following significant precipitation events through May 2022 and analyzed for pH and specific conductance. Ion composition was also monitored periodically (only 2021 data reported here).

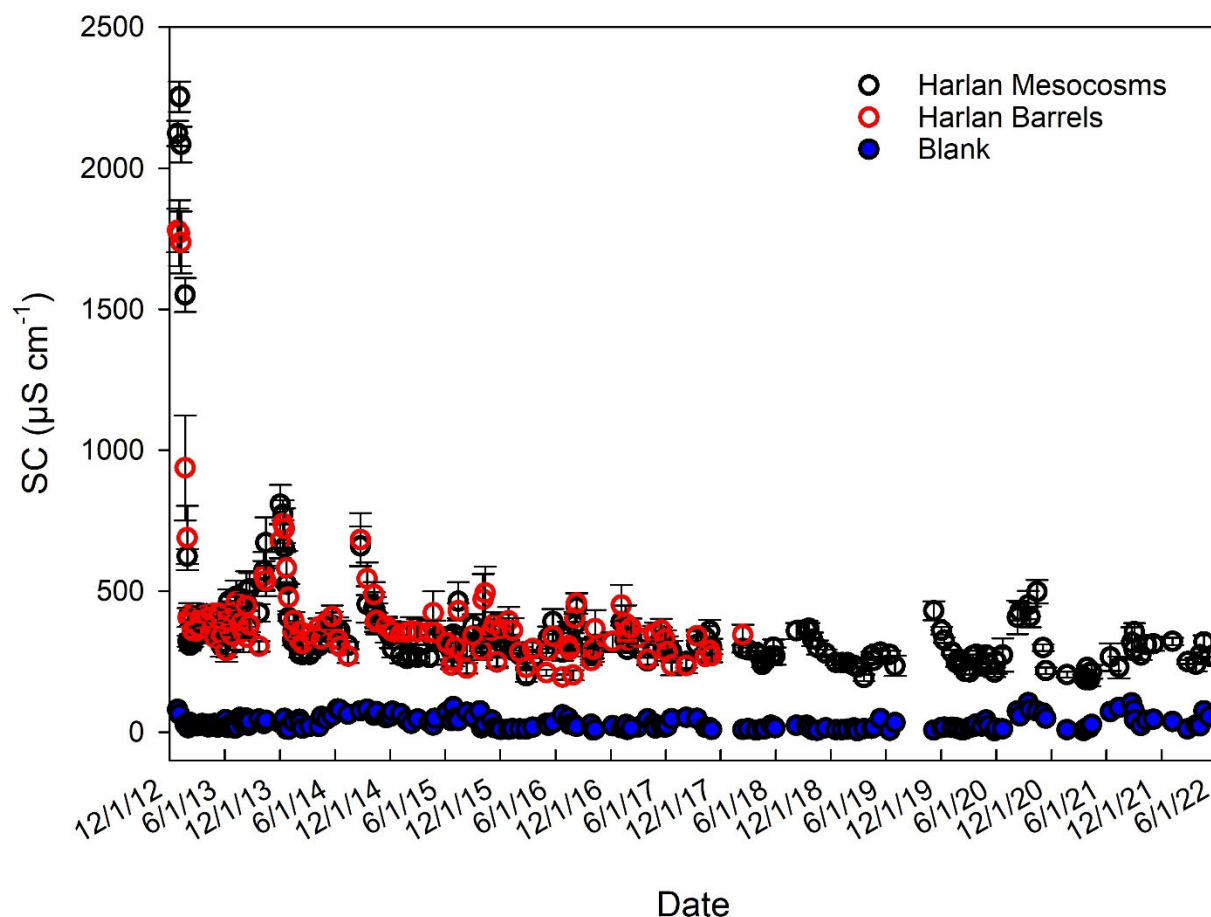


**Figure 3.** Leachate pH over time (through May 2022) for Harlan mesocosms and barrels. Blank refers to an onsite, and presumably clean, barrel collecting ambient rainfall. Mesocosm leachate volumes (not shown) were lower due to evaporative losses and internal water detention in the spoils over time vs. blanks. Values shown are averages ( $n = 3$ ) with one SE above/below.

We periodically analyzed leachate samples for their full suite of soluble ions contributing to SC and example data for one of our 2021 sample events are presented in Tables 1 and 2. The ten-year field leachate specific conductance (SC) data (**Fig. 4**) indicates that (a) leachate EC/SC fell to  $\sim 300 \mu\text{S}/\text{cm}$  by the end of the second full year of leaching, but that (b) the seasonal winter increase continued to occur through 2018, with lower amplitude each year. However, it is interesting to note that the winter high SC (**Fig. 4**) appeared to be increasing slightly between 2019 and 2021 relative to previous years. This probably indicated the onset of more rapid acid metal hydrolysis reactions (Fe and Mn) as the spoils continued to weather and leach, while at the same time neutralizers were depleted and pH dropped (as discussed above). It is important to note that the mesocosms have been kept unvegetated, so organic matter turnover reactions are not responsible for any changes in pH or SC. A more detailed view of the combined 2020 and

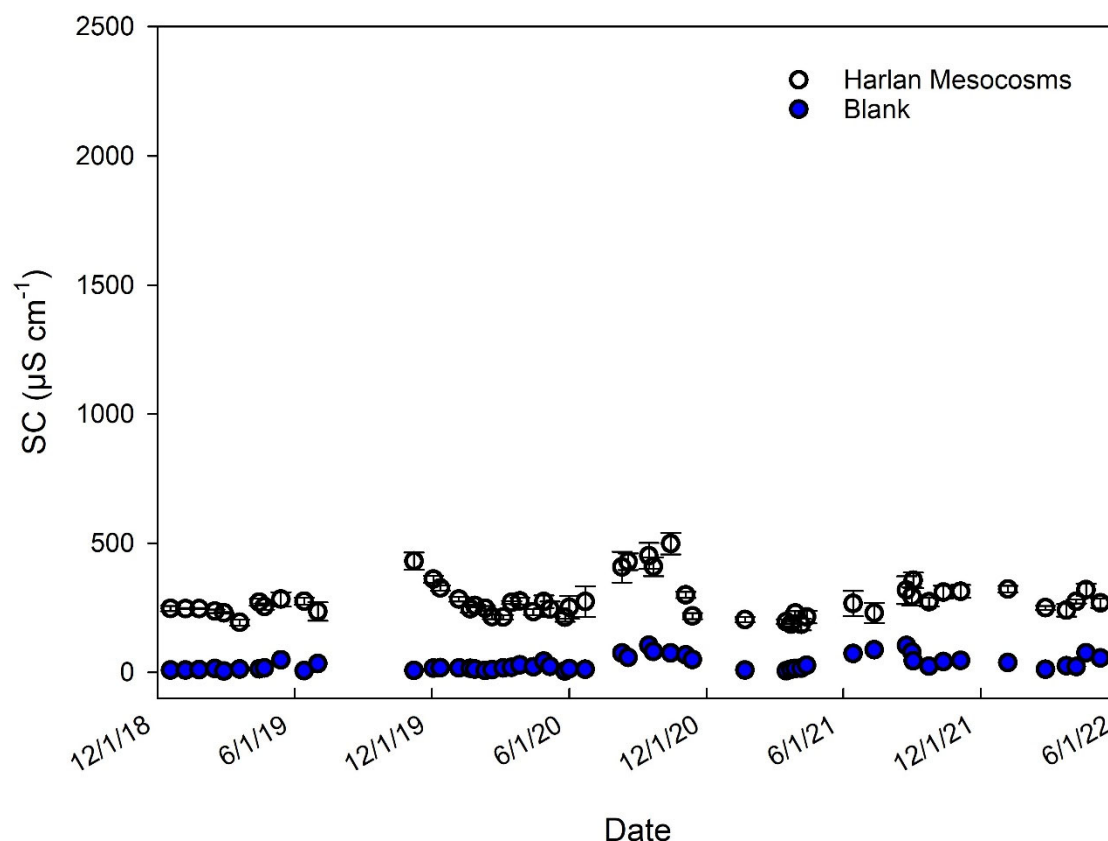
2022 calendar year data sets (**Fig. 5**) indicates that the amplitude of the “seasonal fall bump” in SC was much lower in 2021 and that SC appears to be slightly increasing again over time.

The very high ( $> 1000$   $\mu\text{S}/\text{cm}$ ) initial SC data are due to release of sulfate salts driven by rapid initial oxidation and hydrolysis of trace sulfides in the spoils. With time, we (Orndorff et al., 2018) we have shown that after this initial “weathering flush”, the counter anion complement changes from sulfate to bicarbonate control. The seasonal spike (high in winter) in SC is due to the accumulation of weathering products (both sulfates and carbonates) on spoil surfaces over the dry summer months that are then released the following winter when evaporation losses drop. We have also observed this in a number of field settings, particularly coal refuse scenarios.



**Figure 4.** Leachate specific conductance (SC) over time (through May 2022) for Harlan mesocosms and barrels. Blank refers to an onsite, and presumably clean, barrel collecting ambient rainfall. Mesocosm leachate volumes (not shown) were lower due to evaporative losses and internal water detention in the spoils over time vs. blanks. Values shown are averages ( $n = 3$ ) with one SE above/below.

The relatively large increase in SC over the first full winter leaching season (2013/2014) may have indicated that mechanisms other than simple CO<sub>2</sub> partial pressure and carbonate/bicarbonate equilibrium may be involved. It is likely that highly reactive feldspars and other oxidizers and hydrolyzed mineral surfaces generated reaction salts that accumulated over the drier preceding summer months (Clark et al., 2018). These salts then dissolved and were flushed from the system under much wetter winter leaching conditions. This process would also account for the diminishing “winter flush” observed over time as highly reactive phases were consumed.



**Figure 5.** Detail of leachate specific conductance (SC) between 2018 and 2022 for Harlan mesocosms and barrels. Blank refers to an onsite, and presumably clean, barrel collecting ambient rainfall. Mesocosm leachate volumes (not shown) were lower due to evaporative losses and internal water detention in the spoils over time vs. blanks. Values shown are averages ( $n = 3$ ) with one SE above/below.

**Table 1.** Metals data from January 21, 2021. M1, M2, and M3 refer to samples collected from the different mesocosm replicates, and blank refers to samples collected from the tank that did not include mine spoils.

		<b>M1</b>	<b>M2</b>	<b>M3</b>	<b>Blank</b>
<b>Al</b>	ug/L	11.0	15.6	10.4	9.3
<b>As</b>		0.1	0.1	0.1	0.1
<b>Cd</b>		BDL*	BDL	BDL	BDL
<b>Cu</b>		0.6	0.9	0.8	0.4
<b>Fe</b>		8.8	8.8	6.9	3.2
<b>Mn</b>		0.7	1.7	30.2	4.0
<b>Ni</b>		0.2	0.1	0.19	0.2
<b>Pb</b>		0.1	0.1	0.1	0.1
<b>Se</b>		0.2	0.1	0.2	BDL
<b>Zn</b>		2.3	3.7	3.0	2.5
<b>Ca</b>	mg/L	25.51	19.89	27.49	0.325
<b>K</b>		3.2	3.1	3.02	0.292
<b>Mg</b>		13.14	11.71	12.55	0.156
<b>Na</b>		0.238	0.186	0.215	0.282
*Below detection limit (0.1 ug/L)					

**Table 2.** Total-S and IC data from January 21, 2021. M1, M2, and M3 refer to samples collected from the different mesocosm replicates, and blank refers to samples collected from the tank that did not include mine spoils.

	<b>M1</b>	<b>M2</b>	<b>M3</b>	<b>Blank</b>
<b>Inorganic carbon (mg/L)</b>	21.7	18.2	20.8	1.6
<b>SO<sub>4</sub> (mg/L)</b>	16	16	21	2.1

Finally, the fact that the long-term average SC for the mesocosm leachates dropped below 350  $\mu\text{S}/\text{cm}$  over time has important regulatory implications. Current federal/state guidance and related PRP research findings (Timpano et al., 2018) for TDS release from Appalachian coal mines indicates that 350  $\mu\text{S}/\text{cm}$  is an important threshold for limiting impacts to aquatic macroinvertebrates. That being said, recent increases (minor but more variable) in late fall/winter leachate SC may continue to temper that finding.

The overall short-term lab column (Parker, 2013; Ross, 2015) results combined with the ten-year field barrel and mesocosm scale data offers a unique and unprecedented opportunity to assess both scaling effects and temporal dynamics of TDS release by actively weathering high pH spoil materials. Over the next year, we will continue to compare them with the periodic elemental leachate data to produce and estimate of overall elemental release/depletion rates along with an assessment of the ability of the initial rapid column method to accurately predict longer term field leaching behavior.

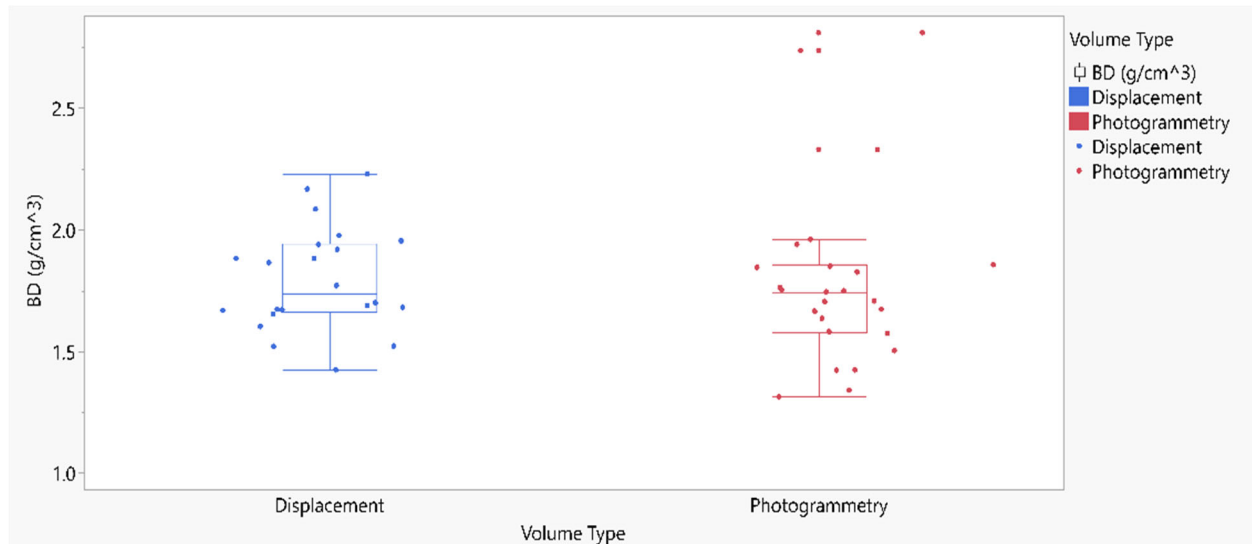
#### *Laboratory characterization of mesocosm spoils*

We analyzed peds ranging from 7 to 450  $\text{cm}^3$  for bulk density using two methods – a traditional paraffin-coated displacement measurement and a much faster method we developed using photogrammetry – and determined that most peds had bulk densities between 1.6 and 1.9  $\text{g cm}^{-3}$  (**Fig. 6**). We found that the photogrammetry-based volumetric scanning method yielded an average bulk density that was statistically indistinct from the traditional paraffin-wax displacement method. This conclusion is based on a Fisher LSD and Tukey HSD test that found no significant difference in means ( $\alpha = 0.05$ ). Despite the overall similarity between these two methods, the photogrammetry method did exhibit several outliers that overestimated bulk density due to underestimated soil clod volume. These underestimations of soil clod volume arose during conversion of the raw point cloud into a closed mesh geometry. It is recommended that multiple volumes from separate point clouds of a single clod be averaged to control for raw 3D data of varied quality.

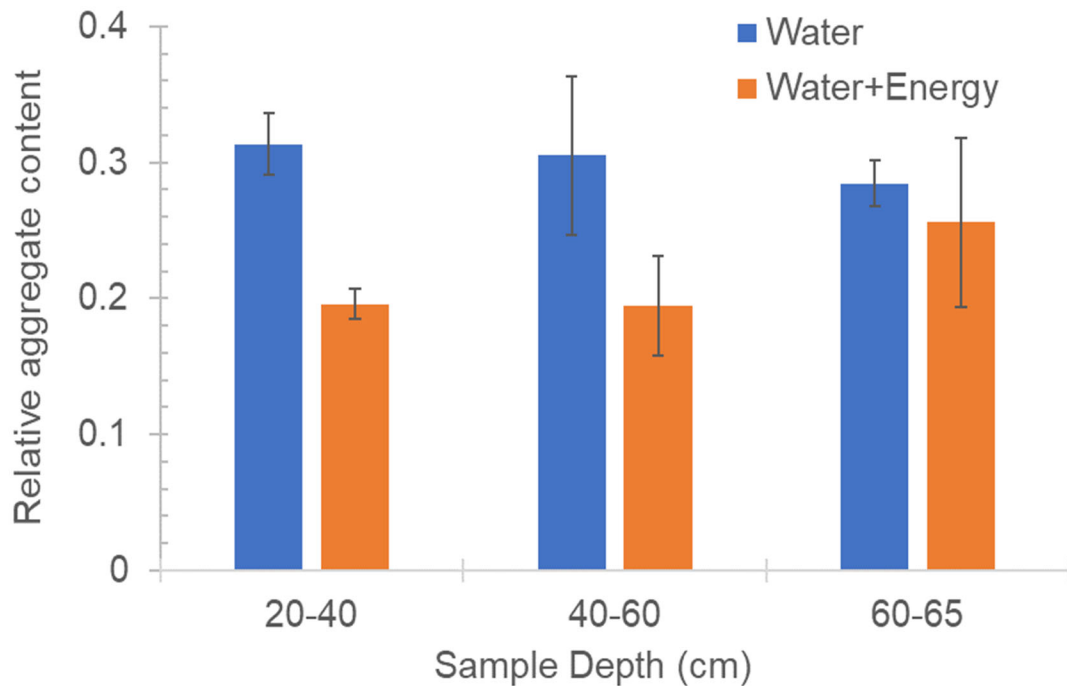
Our particle and aggregate size analyses indicated that the  $< 2$  mm size fraction was moderately aggregated (**Fig. 7**). When the samples were dispersed using only water, approximately 0.3 (30%) of the particles were aggregated, with little differences between the three depth increments. When ultrasonic energy was applied to further disperse the aggregates, the deepest samples (60-65 cm) had a mean of 26% aggregated particles, compared to  $< 20\%$  for the 20-40 and 40-60 cm depths. This result suggests that the aggregates near the bottom of the mesocosms were less resistance to disturbance, possibly due to incipient cementation. It is not known if the lower boundary influenced this particular result, so future work will examine aggregate stability from reconstructed soil profiles in field settings (e.g., the COP plots).

The LIBS scanning revealed differences between the exterior and interior elemental intensities (**Fig. 8**). The raw LIBS spectra of representative scans of a single sample depict an overall

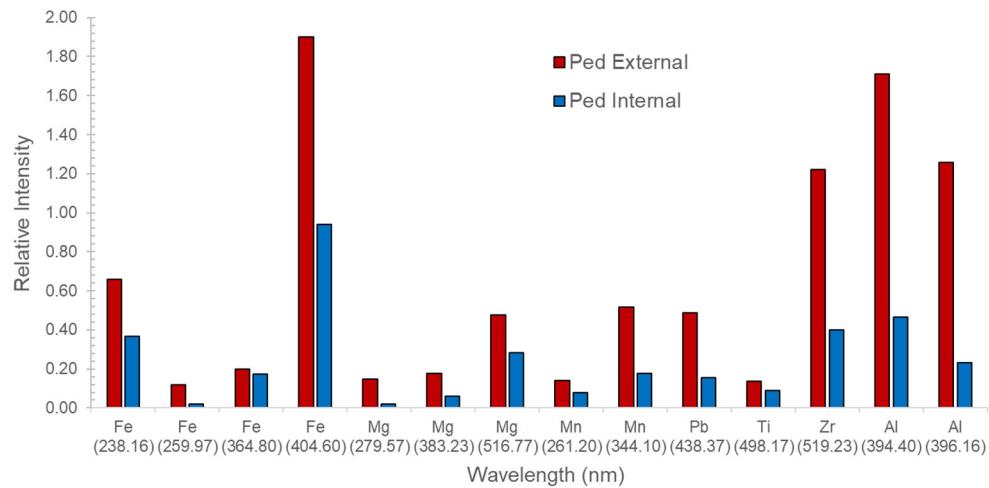
denudation of intensity of the ped exterior relative to the interior from ~300 to 700 nm (**Fig. 1**). Conversely, from ~190 to 300 nm, peak signal intensities were greater in the ped interior, suggesting that there are spatio-elemental differences in soil aggregates. When the signal denudation effect was controlled for by dividing the raw intensity value for each wavelength by the sum of the total spectra intensities, peak intensity values for metals (Fe, Mg, Mn, Pb, Ti, and Al), were greater on the ped exterior across multiple wavelengths (**Fig. 8**). These findings suggest that metal ions or oxides are becoming concentrated at the ped exterior, possibly due to 1) internal transport processes, wherein metals are being drawn to ped surfaces, for example due to small-scale wetting and evaporation processes at ped surfaces, 2) armoring effects, where the presence of those elements help define ped boundaries (e.g., by providing better resistance to disaggregation), or 3) spatially-distinct redox conditions driving mineral dissolution and re-crystallization processes (Zuasing et al., 2013).



**Figure 6.** Bulk densities of intact peds collected from the mesocosms ( $n = 47$ ). Bulk densities were measured using (left) water displacement of paraffin-coated samples and (right) photogrammetry. Boxes indicate 25<sup>th</sup>-75<sup>th</sup> percentiles and whiskers indicate 5<sup>th</sup>-95<sup>th</sup> percentiles.



**Figure 7.** Aggregate content of samples from the TRC mesocosms. Blue shows samples dispersed in water only; orange indicates dispersion by water and ultrasonic energy.



**Figure 8.** Relative intensity from LIBS spectra for wavelengths associated with different metals in the exterior (red) versus interior (blue) of an example ped.

## **Conclusions**

This report presents field and laboratory data associated with a long-term mesocosm study. The mesocosms were filled with a typical southwest Virginia sandstone mine spoil (material from the Harlan formation) and allowed to weather over 9.5 years. The combined results from our previously reported COP column leaching studies and the mesocosm data confirm that our lab column protocol appears to be a relatively accurate predictor of peak vs. long-term leachate specific conductance (SC; proxy for TDS), though it probably under-predicts peak seasonal ion release events in younger reactive materials. It is also notable that even though analyses of the mesocosm leaching data over the first 7 years indicated a consistent and gradual overall decline in TDS elution to levels lower than the presumed 350  $\mu\text{S}/\text{cm}$  critical level for instream biotic effects, more recent data collected in 2021 and 2022 indicates the potential for a subsequent minor rebound in SC along with associated lower pH levels.

The deconstruction of the Harlan mine spoils in the TRC mesocosms provided the opportunity to gain new insight into aggregation processes. During deconstruction we collected and analyzed intact soil aggregates and clods for their size, bulk density, and elemental composition. We analyzed samples from the following depth increments – 0-20, 20-40, 40-60, and 60-65 cm – from each of the three mesocosms. Elemental composition was quantified using a handheld laser-induced breakdown spectroscopy (LIBS) scanner. We tested homogenized samples of 1) large clods and 2)  $<2$  mm aggregates, and compared them to 3) unaggregated bulk soil, and then analyzed spatial variability of elemental concentrations within aggregates by splitting large clods into surface versus interior subsamples.

Those analysis revealed that the mine spoils had formed many large peds (i.e.,  $> 1\text{-}2$  cm in diameter) as well as smaller aggregates (i.e.,  $< 2$  mm in diameter). Larger peds had measured bulk densities that were mostly between 1.6 and 1.9  $\text{g cm}^{-3}$ , which is in the range of expected values based on other work. It remains unknown whether this internal consolidation and aggregation may help to explain the auto-compaction observed in reconstructed mine spoil profiles such as those of the COP plots. The elemental scanning work suggest that metal molecules may be more prevalent near the external surfaces of peds, where those elements may help resist disaggregation. Small aggregates collected from the bottom of the mesocosms also had greater resistance to disaggregation, which may imply an incipient cementation process was occurring. Future work will use similar methods to assess whether similar processes are occurring within larger-scale reclamation sites.

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