# Predicting Plant Metal Bioaccessibility at the Historic Wheal Maid Tailings Lagoons, Cornwall, UK

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**Abstract** Abandoned mine sites with their metal-rich substrates pose significant challenges to naturally colonizing plants. In this study, the abandoned Sn-Cu tailings lagoons at Wheal Maid (Cornwall, UK) have been investigated to establish the bioaccessibility of metals and metalloids (As, Cd, Cu, Pb, Sb, Zn) in exposed tailings and wastes using a new plant bioaccessibility test. Four main substrate types were sampled: (1) mine waste used to construct the lagoons, a relatively uncontaminated material with variable particle size; (2) granular capping material used in the upper lagoon to cover the tailings and relatively uncontaminated; (3) grey tailings a fine to medium grained material with visible sulfides and white secondary salts, extremely high in near-total Zn concentrations; and (4) marbled tailings a fine grained brown/red/yellow material with extremely high near-total As concentrations. The analytical quality of results produced by a new plant bioaccessibility test was monitored using blanks, spiked solutions and repeat analyses. The grey tailings had the highest bioaccessible metal and metalloid content. As this material oxidizes, it will release As, Cd, Cu, Sb, Zn and to a lesser extent Pb in a form which will be more available to plants. This will inevitably delay re-vegetation at the site. The new bioaccessibility test is recommended for sulfidic rocks and waste samples and

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should be employed at an early mine-life stage to allow appropriate waste classification and improve mine closure outcomes.

## Introduction

Abandoned mine sites undergo natural colonization by plants (Lottermoser, 2011; Jiménez 2011). Establishment of a sustainable post-mining vegetation is essential for successful mine site rehabilitation and should be planned for at an early mine-life stage. To predict re-vegetation processes and challenges a thorough knowledge of the substrate is required. Characterization of the bioaccessibility of potentially toxic trace elements in mine site substrates provides useful data to help forecast re-colonization. This can be achieved using sequential extraction tests to define the geochemical fractionation of these elements (e.g. Al-Abed et al. 2006; Dold 2003; Khorasanipour et al. 2011). However these tests are lengthy and therefore expensive to preform, prohibiting their routine application. This study presents the results of a case study using a new test to determine the plant bioaccessibility of trace elements in sulfidic wastes and rocks. The test is rapid, repeatable and inexpensive. It can be used to obtain operational plant bioaccessibility data for mine site samples on a routine basis. This will improve environmental risk assessments and hence waste management plans and mine closure outcomes at these sites.

## The Wheal Maid Site

### *Location and Physiography*

The Wheal Maid site is located in the Gwennap Mining district and forms part of the Cornwall and West Devon Mining Landscape, which has been on the UNESCO world heritage list since 2006. The Gwennap Mining district is one of the most intensively mined in the Cornwall and Devon Sn and Cu mining province. During the 18th and 19th century this was the richest mining district in Cornwall and was referred to by contemporary writers as “the richest square mile to be found anywhere on the earth” (Cornish Mining 2015).

Approximately 10 km northwest of Falmouth, the site has its center at 50°14′14.61″N and 5°9′37.85″W. The site comprises two lagoons which are bordered by a path which runs around the perimeter of the site. The western lagoon is referred to as the upper lagoon and has some vegetation cover and some standing water (Fig. 1). The eastern lagoon is referred to as the lower lagoon, this has no vegetation and the standing water is a striking orange colour (Fig. 2) due to the Fe oxides present in the water.

**Fig. 1** Granular capping at the upper Wheal Maid tailings lagoon, with evidence of some re-vegetation (foreground) and mine waste used to construct the lagoons (beyond the standing water, middle distance) (color figure online)

**Fig. 2** Marbled tailings (foreground) and standing water at the lower Wheal Maid tailings lagoon (color figure online)

## *Geology*

Mineralization in this area is largely hosted by a series of upper Devonian metamorphosed sediments (dominantly shales) known locally as “killas”. Deformation of the sediments occurred prior to and during the period of granite intrusion with later minor deformation. The major period of deformation took place during the Variscan orogeny (mountain building) and in the later part of this orogeny the granites were intruded with associated tin and copper mineralization, forming the Cornubain Orefield which covers the whole of the county of Cornwall and part of the county of Devon (Camm and Hedley 2005).

Most of the tailings in the lagoons were deposited as waste from the nearby Mount Wellington mine during the late 1970s (Freeborn 2006). The mineralogy of the lodes exploited by this mine is therefore relevant when evaluating the environmental impact of those wastes decades later. The Mount Wellington mine lies approximately 2 km south east of the Wheal Maid site, approximately 3 and 4 km from the nearest outcrops of the Carnmarth and Carmenellis granites. Kettaneh and Badham (1978) detailed the mineralization and paragenesis at the Mount Wellington Mine at a time when the mine was still operational. They highlight six lodes in the mine; numbers 1, 2 and 3 and the Wheal Andrew, the Trenares and the Hot lodes. The main lodes were numbers 1, 2 and 3 which produced Sn, Cu and Zn concentrates and contained Fe, Zn, Sn, As, Cu, Ti and lesser Pb, Ag, Au, W, Bi and Sb. The mineralization in these three lodes was very similar and the bulk of mineralization is accounted for by two main phases. Initially following emplacement and cooling of the intrusion, extensive fracturing permitted the introduction of mineralizing fluids in phase A; these fluids precipitated considerable amounts of quartz, tourmaline and cassiterite with minor amounts of wolframite. Renewed movements in the fractures permitted the introduction of phase B where quartz, chlorite and sulfides are abundant. No cassiterite was introduced and there is therefore a dilution of the tin grade in this phase. During phase B, chlorite, quartz, pyrrhotite, arsenopyrite and chalcopyrite were the first minerals precipitate. These were followed by more quartz with sphalerite (containing exsolved chalcopyrite), rutile, minor galena and probably small grains of gold. The phase ended with the precipitation of large amounts of pyrite which overgrew earlier minerals and cemented the rock. It is unclear when the secondary marcasite and covellite observed in this association formed.

## *Climate and Vegetation*

The local climate is influenced by its position on the southwest peninsular which forms the most southerly part of the British Isles. There is strong maritime control of temperatures and annual averages are close to the surrounding sea temperatures at 11–12 °C. Frost frequency is highly variable and the region has a reputation for mildness. Rainfall totals are approximately 900–1000 mm and the area is prone to rare but heavy rainfall events lasting from 5 to 15 h (Met Office 2015).

Plant cover around the site is patchy and the extent of re-vegetation of the upper and lower lagoons is very different. Very little re-vegetation has occurred over the lower lagoon, there is no vegetation present on the exposed grey or marble tailings although some *Calluna vulgaris* (heather) has begun to colonize the periphery. This is in contrast to the granular capping of the upper lagoon which was seeded in the 1980s. The area of the upper lagoon which is slightly elevated and distant from the standing water appears to have benefited from the remedial measures and shows a variety of vegetation. Colonizers at the site occur mainly at the margins of the mine waste constructed paths around the lagoons and include predominantly *Calluna vulgaris* (heather) and *Ulex europaeus* (gorse) which have the ability to exclude metals and arsenic from their above ground biomass (Lottermoser et al. 2011) and are often found at abandoned mine sites because of this. Outside the perimeter path of the lagoons grow *Pinus radiata* (Monterey Pine) trees, a native of rocky headlands and islands on the Pacific coast of California. This salt tolerant tree is often planted as an amenity or shelter plant and has the ability to tolerate harsh conditions. There are records of them being planted at the nearby United Mines, St. Day in the early 1980s (CC 2015) and it is likely that these larger specimens date from that period. In addition to trees that have likely been planted, there are some younger plants that may well have self-seeded.

## Site History

The tailings lagoons were built in two separate construction phases between 1976 and 1981 and contain approximately 220,000 $m^3$ of fine grained mineral processing waste. The downstream and intermediate embankments were constructed during the first stage between 1976 and 1978 whilst the land was under the ownership of Cornwall Tin and Mining Ltd. This mining company used the site to dispose of mill tailings from the nearby Mount Wellington mine by pumping them along the valley in slurry form. Part of phase one also involved diverting the Wheal Maid stream into a 1200 mm concrete pipe which ran beneath the depository for approximately 440 m. After Billiton Minerals purchased the surface plant at Mount Wellington mine and the Wheal Maid tailings dams in 1979, an upstream embankment and a further 396 m of culvert were constructed. Material from the downstream dam, along with other material from the Carnon Valley, were reprocessed, and the reworked deposits were pumped into the upstream dam. Mount Wellington mine closed in 1981. In 1982 Carnon Consolidated Tin Mines Ltd. purchased Billiton's interest including the Wheal Maid site and no further waste was deposited in the dams. An attempt was made at the time to prevent wind-blown dust by trial seeding of the upper dam, which was partially successful with the growth of a variety of vegetation across the dam surface (Freeborn 2006; Gwennap 2015).

Gwennap Council now owns the site (CDC 2008), having purchased the site in 2002 for £1 from Carnon Enterprises. There is no right of access onto the site, although it is freely accessible via a footpath on the western boundary and a section of the mineral tramway footpath runs along the southern boundary. The site is regularly used by walkers, mountain bikers and horse riders, also on occasion by motorcyclists and four wheel drive vehicles, although this is not permitted. In 2013 a section of the perimeter formed part of the course for the British Cycling National Cross Country MTB competition.

## *Previous Investigations*

A record of determination of the Wheal Maid tailings lagoons as contaminated land was made by Carrick District Council in 2008 (CDC 2008) due to the risk of significant harm to young children using the site and the risk of pollution of controlled water through leaching of potentially toxic trace metals and metalloids (As, Cd, Cu, Cr, Fe, Pb, Ni and Zn). The evidence on which the determination was based is detailed in the record of determination (CDC 2008) and includes: (a) monitoring data from the Environment Agency suggesting the site could be causing a failure of Environmental Quality Standards (EQSs) in the St. Day Stream; (b) a desk study to establish a conceptual model for the site; and (c) an intrusive investigation carried out in 2007 which undertook soil, groundwater and surface water sampling and analysis. The four main soil samples identified during the intrusive investigation were:

1. Mine Waste (MW). This material was used in the construction of the dams and is located around the perimeter of the lagoons. It is a granular material with a variable particle size (Fig. 1).
2. Granular Capping (GC). This material was used in the upper lagoon to cover the tailings; some of it was subsequently seeded in an attempt to remediate the land (Fig. 2).
3. Marbled Tailings (MT). This material was located around the lower lagoon. It is a fine grained brown/red/yellow material (Fig. 2).
4. Grey Tailings (GT). This material was located at one end of the lower lagoon. It is a fine to medium grained material (Fig. 3). Grains of sulfide material can be seen by eye and there are white effervescent salts evident on the surface of the material in some places.

Variable high metal and metalloid concentrations were reported for all four materials (CDC 2008). A water leachate test showed that the proportion of metals and metalloids in the four materials upon a heavy rainfall event was very different. Leach testing of the grey tailings showed that there was a high leaching potential for As, Cd, Cu, Pb, Ni and Zn, and a likelihood that the exposure, erosion and weathering of the pyritic grey tailings in the lower lagoon contribute to the very poor, acidic water

**Fig. 3** Grey tailings at the lower Wheal Maid tailings lagoon (color figure online)

quality within the lower lagoon and the St. Day Stream, to which seepage occurs at the lower end of the retaining dam. The leachable metals in the mine waste and granular capping material were generally Cu and Zn with some Cr. In the marbled tailings, As concentrations were high, however As did not appear to be readily leached. Cu and Zn did leach from the marbled tailings but to a lesser extent than from the other materials on the site. Of the four materials, the grey tailings were considered to pose the most significant contamination risk (CDC 2008).

## Mineralogy and Geochemistry

Forty one bulk surface samples (ca. 500 g) were collected from the Wheal Maid site. These were grouped by the four different sample types: mine waste (MW), grey tailings (GT), marbled tailings (MT), and granular capping (GC). Samples were dried and sieved to <2 mm. At ALS laboratories, the samples were subsequently pulverized to 85 % <75 μm, aliquots of the sample were then digested with perchloric, nitric, hydrofluoric and hydrochloric acid and analyzed using ICP-AES. Analysis included replicate analyses of the NIST 2710a Montana soil I standard reference material, for which the relative standard deviations of analysis were <5 % and recoveries were in the range 90–110 %. The mean geochemistry (± standard deviation) for the major and environmentally relevant trace elements was calculated for each of the four main soil types and is given in Table 1 below.

Concentrations of As are particularly high in the marbled tailing samples. Positive correlation coefficients of the log normalized Fe and S concentrations suggest that arsenopyrite (FeAsS) is the source of these elevated concentrations. Copper is elevated in both the granular capping and grey tailings. Cu correlates well with S and Zn in the granular capping. This possibly indicates an association between sphalerite and chalcopyrite as was suggested by Kettaneh and Badham (1978). There is no obvious single mineral responsible for Cu in the grey tailings; when all the samples are considered the only correlation observed is between Cu and In. However, when only the five high copper grey tailing samples are considered, correlations with Zn and Cd but also to a lesser extend Pb and S become more important. This also possibly supports the sphalerite/chalcopyrite association suggested by Kettaneh and Badham (1978). The Zn in the grey tailings correlates only with Cd when all samples are considered, however, if only the five samples with the highest Zn concentrations are considered the correlation with Fe and S increase, indicating that sphalerite is the likely source of Zn in the high zinc grey tailings samples.

The multi-addition NAG technique (mNAG) (Smart et al. 2002) involves three cycles of addition of 15 % $H_2O_2$, effervescence and heating. The results (Table 1) give an indication of the acid generating capacity of the sample when sulfides within the sample become oxidized over time. The mNAG pH is lowest in the grey tailings, as this is a substrate type with visible pyrite in many of the collected samples. However, the mean $CaCl_2$ paste pH is lower in the marbled tailings indicating that the sulfides have undergone some oxidation, releasing immediate acidity rather than requiring oxidation of the sulfides to effect the acidity. Both the grey and marbled tailings have a lower pH than the mine waste and granular capping materials.

## Bioaccessibility Testing

The test for metal and metalloid bioaccessibility as described previously was applied to each of the 41 samples (<2 mm) collected from the Wheal Maid site. In a similar method to the mNAG, the samples were reacted with hydrogen peroxide, heated, evaporated and the cycle was repeated three times. The resultant soluble metals and metalloids in the moist sample were then extracted using ammonium acetate solution and submitted for analysis at ALS, Ireland. Analytical quality of the test was monitored by the inclusion of blank extracts (determinations all <2 × LOD), spiked solutions (recoveries 85–115 % except for Sb (78 %)) and replicate analyses of a single sample (relative standard deviations all <10 %). The results are presented in Table 2.

Some of the detected metals and metalloids are contained in sulfide minerals. However, following oxidation of the material these elements will become available

**Table 1** Mean and standard deviations for selected elemental concentrations and pH values determined for the four sample types

| Mean value (standard deviation) by material type | | | | |
|---|---|---|---|---|
| | MW (n = 10) | GC (n = 10) | GT (n = 10) | MT (n = 11) |
| Major elements (wt%) | | | | |
| Al | 8.31 (0.94) | 7.11 (0.87) | 3.58 (1.43) | 4.98 (1.24) |
| Ca | 0.12 (0.14) | 0.11 (0.05) | 0.04 (0.01) | 0.04 (0.02) |
| Fe | 5.9 (0.7) | 5.8 (1) | 15.4 (8.2) | 20.3 (4.5) |
| S | 0.31 (0.25) | 0.12 (0.09) | 6.4 (1.13) | 2.44 (0.56) |
| Minor elements (ppm) | | | | |
| As | 1081 (653) | 2470 (1010) | 2332 (1260) | 13,325 (5350) |
| Cd | 0.43 (0.32) | 1.6 (1.27) | 18.8 (22.5) | 0.88 (0.57) |
| Cu | 378 (202) | 893 (577) | 694 (755) | 141 (57) |
| Pb | 130 (53) | 416 (136) | 395 (258) | 304 (109) |
| Sb | 8.8 (5.7) | 13.9 (5.2) | 12.6 (5.9) | 13.4 (3.2) |
| Zn | 271 (81) | 399 (287) | 11,590 (16,790) | 364 (319) |
| pH measurements | | | | |
| mNAG | 4.08 (0.98) | 6.55 (0.45) | 1.93 (0.12) | 2.63 (0.11) |
| Paste $CaCl_2$ | 4.11 (0.99) | 4.46 (0.25) | 2.58 (0.36) | 2.42 (0.11) |

mine waste (*MW*), granular capping (*GC*), grey tailings (*GT*) and marbled tailings (*MT*)

**Table 2** Bioaccessibility of metals and metalloids in MW, GC, GT and MT samples expressed as a percentage of the total element concentrations

| Mean value (standard deviation) by material type | | | | |
|---|---|---|---|---|
| Element (%) | MW (n = 10) | GC (n = 10) | GT (n = 10) | MT (n = 11) |
| As | 2.4 (1.6) | 2.8 (2.5) | 60.2 (12.1) | 1.4 (0.4) |
| Cd | nr | nr | 65.7 (9.8) | nr |
| Cu | 33.9 (14.9) | 42.3 (15.8) | 55.6 (12.6) | 38.7 (13.5) |
| Pb | 3.9 (2.4) | 6.7 (6.6) | 18.1 (22.9) | 0.6 (0.2) |
| Sb | nr | nr | 35.1 (13.0) | nr |
| Zn | 16.9 (13.9) | 13.2 (15.3) | 57.5 (20.3) | 48.7 (19.5) |

*nr* not reported because a high number of results for the bioaccessible fraction were below the limit of detection

to plants either planted on, or colonizing the substrate. The hydrogen peroxide in the bioaccessibility test accelerates the oxidation process and the ammonium acetate solubilizes the elements liberated by this accelerated oxidation.

All results for As are above the LOD for the bioaccessibility liquior and it is clear that As is particularly bioaccessible (62 %) in the grey tailings. Arsenic in the marbled tailings is far less bioaccessible (1.4 %) since there is a much larger reservoir of total As in these samples.

Many of the results for the determination of Cd in the bioaccessibility leachate were less than or near the limit of detection. However, the bioaccessibility of Cd in the grey tailings seemed to be consistently high, particularly in two samples of the white precipitate present on the surface of the grey tailings which had a Cd bioaccessibility of 74 and 84 %. The grey tailings is the sample set with the highest total concentration of Cd, and the log normalized concentrations correlate highly with those of Zn.

Cu bioaccessibility is consistently high in all four sample types, although slightly higher in the grey tailings. Lead in the grey tailings was found to be the most bioaccessible (18 %). However, the standard deviation for this figure was relatively large (23 %), indicating high variability in the bioaccessibility of Pb in these samples. Lead concentration in the white precipitate associated with the samples is very low, and when these samples are excluded, the Pb bioaccessibilities range from 2.6 to 70 %.

The bioaccessibility analysis for Sb suffered from the same limitation as Cd in that many of the results for the bioaccessibility liquor were below or close to the limit of detection. However, it is interesting that for Sb whilst the grey tailings were the only samples with a measureable bioaccessibility of 35 %; these were not the samples with the highest total Sb concentrations.

Zinc is particularly bioaccessible in the grey and marbled tailings and is also bioaccessible in the granular capping and mine waste materials. Within the grey tailings, Zn bioaccessibility in the white precipitate is particularly high at 82 and 94 %. These samples also have high total Zn concentrations and will provide a large input of this element into the mobile phase upon oxidation. Apart from these two samples, the grey tailings generally have both high bioaccessibility and total concentrations for Zn. It is likely that the oxidation of sphalerite in these high zinc samples is the source of the bioaccessible Zn.

## Conclusions

The current use of bioaccessibility testing for contaminated land assessment is dominated by the assessment of oral bioaccessibility of potentially harmful elements to humans (e.g. Denys et al. 2012; Ruby et al. 1996; Van De Wiele et al. 2007). Revegetation of historic mine sites is important for successful rehabilitation and can be delayed if environmentally significant elements are elevated and bioaccessible. Sequential extraction techniques (e.g. Dold 2003) determine the association of potentially toxic trace elements with different geochemical fractions and as such can help predict plant bioaccessibility. However, these tests are time-consuming and expensive and are consequently not used by the mining industry on a routine basis (Parbhakar-Fox and Lottermoser 2015). Moreover, they provide detailed but unnecessary information on inert geochemical fractions. In sulfidic wastes and rocks, it is the potentially toxic elements associated within sulfide minerals that are bioaccessible as they may become mobile and available to plants upon oxidation (Amos et al. 2015; Kossoff et al. 2014). A rapid, inexpensive and reliable technique to determine this fraction is therefore required before plant bioaccessibility measurements will become fully integrated into mine site risk assessments. A protocol for such a test has been presented previously in this monograph and the results of its application in a case study site are presented here.

The Wheal Maid historic mine site is part of the Cornish mining heritage landscape. It lies on the southwest UK peninsular in an area that was heavily mined during the 18th and 19th century. Disposal of sulfidic tailings in two adjacent lagoons in the 1970s and 80s caused the site to become a major source of potentially toxic trace elements and acid water contamination. The walls and walkways of the lagoons were constructed with mine waste. Colonization by pioneer and intentionally seeded plants has begun to occur on the upper lagoon which has been capped with a granular material. The lower lagoon shows no sign of re-vegetation and there are exposed pyritic (grey) tailings and weathered (marbled) tailings. Standing water in the upper lagoon is not visibly contaminated but in the lower lagoon it is a striking orange colour due to the presence of Fe oxides in the water.

Geochemical analysis shows that the marbled tailings have extremely elevated near-total levels of As whilst the grey tailings are extremely elevated in near-total Zn. Near total concentrations for other elements and materials are of approximately the same order of magnitude, apart from S which is elevated in both the grey and marbled tailings and Cd which is elevated in the grey tailings. Results for the new bioaccessibility test reveal that As bioaccessibility in the marbled tailings is relatively low. It is the grey tailings that contain the most bioaccessible As, Cd, Cu, Sb, Zn and to a lesser extent Pb.

Weathering and oxidation of the grey tailings at the Wheal Maid case study site will continue to release the bioaccessible elements present in this material into a potentially available form for plant uptake. If no remedial action is taken, mine site rehabilitation reliant on a sustainable vegetation cover will inevitably be delayed. The application of the new bioaccessibility test on Wheal Maid samples demonstrate its potential application for other site assessments. In particular, the new test is useful for site scenarios where sulfide oxidation and acid rock drainage are likely.

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