SILVERMINES WETLAND SUBSTRATE REPROCESSING

Prepared for: Environmental Protection Agency

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Small Scale Study: STRIVE Programme

Prepared for: Environmental Protection Agency

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1.0 Introduction

SLR Consulting Limited (SLR) was awarded funding from the Irish Environmental Protection Agency (IEPA) through the Science, Technology, Research & Innovation for the Environment (STRIVE) programme to undertake a preliminary investigation of the metals held in the passive treatment system at the Silvermines mine site, County Tipperary. The purpose of the study was to establish, in principle, if the metals held in the substrate could be extracted as a resource.

1.1 Project Understanding

In a mine water treatment context, passive treatment is a process of removing metals (although inorganic substances such as cyanide, organic nitrogen and phosphorus compounds can also be treated) and/or acidity in a natural-looking, man-made bio-system that capitalizes on ecological, and/or geochemical reactions coupled with physical sequestration. In most cases the process does not require power or chemicals after construction, and can last for decades (or longer) with minimal human intervention.

Man-made passive treatment systems employ the same principles as natural wetlands, but they are designed to optimize the competing processes occurring naturally in a wetland ecosystem.

Such passive systems have a finite treatment capacity before regeneration of the various substrates involved in the treatment is required. These substrates would have collected heavy metals and certain metalloids (depending on the source chemistry) over a period of time such that their concentration in the wetland media could offer a viable economic resource.

From preliminary discussions with EPA, the Silvermines area in County Tipperary, Ireland includes a passive treatment system which is reaching its capacity. These are located in an area associated with a c.74ha tailings facility. The tailings resulted from mining for lead, zinc, copper and barites with some silver. It is likely that zones within it require regenerating through desilting/substrate regeneration. When reconditioning a substrate that is laden with heavy metals there are two realistic options:

1. Disposal as a waste; or
2. Reprocessing/ reusing material.

The key consideration is what to do with the spent substrate material, especially if the metal concentration would classify the material as hazardous from a disposal perspective. It is believed that there are realistic opportunities to examine the economic viability of reprocessing the passive treatment substrate and it is this hypothesis that is the focus of this research.

1.2 Study Objectives

The study aims were to:

1. sample and analyse substrate from the wetland area treating drainage from the Silvermines tailings dam;
2. classify the substrate in terms of waste type should it be excavated for disposal; and
3. Investigate the most environmentally and economically sustainable method of dealing with the substrate.

The intention is to ascertain if reprocessing of the sediment is likely to be viable and whether a resource (such as metals) is a realistic option. The objectives were met by designing a sampling and analysis programme of a preliminary nature, to understand the chemical form of the metal in the substrate.

In addition a number of academic personnel were contacted in Ireland, the US and the UK to understand what wok has been undertaken in this area and if there is any ongoing applicable research. The Environment Agency and IEPA were also contacted to discuss the project. Details of all points of contact are presented in Section 4.

1.3 The Silvermines Site

The Silvermines site in County Tipperary, Ireland comprises an area of approximate 5 square kilometres with an area of historic tailings storage equating to 19 hectares. The area has been mined since the c 17th to 20th century. The main metals which were mined included lead, zinc, copper, silver and barium (in the form of barites).

The area has been subject to a progressive rehabilitation which included the design and installation of a passive treatment system to treat water emanating from an area termed the Garryard Disposal Facility. The design of the treatment systems comprised a settlement lagoon, aerobic and anaerobic wetlands designed to remove the metals leaving the disposal facility. The location of the wetland treatment system is shown below in Figure 1.[1] And Figure 2[2]

SLR has not seen the information which has been used to design the system or the 'as built' drawings of the systems. Notwithstanding the best practise for using wetland systems is described below:

- Aerobic Wetlands. So termed because they encourage the oxidation of the water flowing within and over the wetland substrate. The use of reeds such as Typghus and Phragmites help to main the oxidising environment. To this end such wetlands are usually restricted to the removal of metals which precipitate when they oxidise. Consequently aerobic systems are usually used for the removal of iron and work most efficiently in net alkaline waters. In some cases arsenic (adsorbed to the iron), manganese (at alkaline pH) and non-metals such as cyanide have been successfully removed using such systems.
- Anaerobic wetlands. These rely upon the presence of sulphate-reducing bacteria to reduce the sulphate in the water to sulphide and sequester the metals in the water. The success of such systems is to ensure there are sufficient sulphates reducing bacteria in the wetland substrata. This therefore requires an organic substrate with a source of inoculum (usually manures) to provide the necessary bacterial community.
- The settlement pond at the upstream area of the system would have been used to manage any water flux/surge into the wetland. This helps to prevent overloading and washing away of substrata.

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[1] SRK Consulting (2005) Management and Rehabilitation of the Silvermine Area. Phase IV report: Conceptual Design. Prepared for Department of Communications, marine and Natural resources. Ref: U2465/2

[2] SRK(2011) Silvermine Rehabilitation Project. Project Updated August 2011, PowerPoint Presentation.

Figure 1-1
Silvermines Mine and Wetland Treatment Area

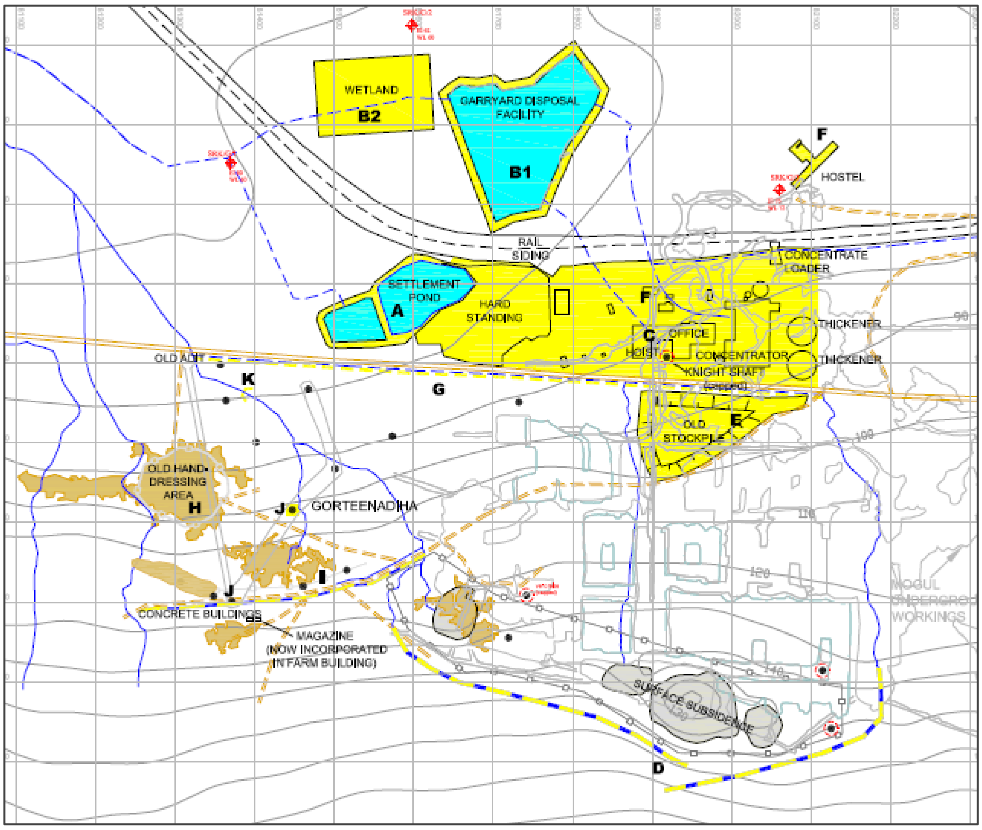
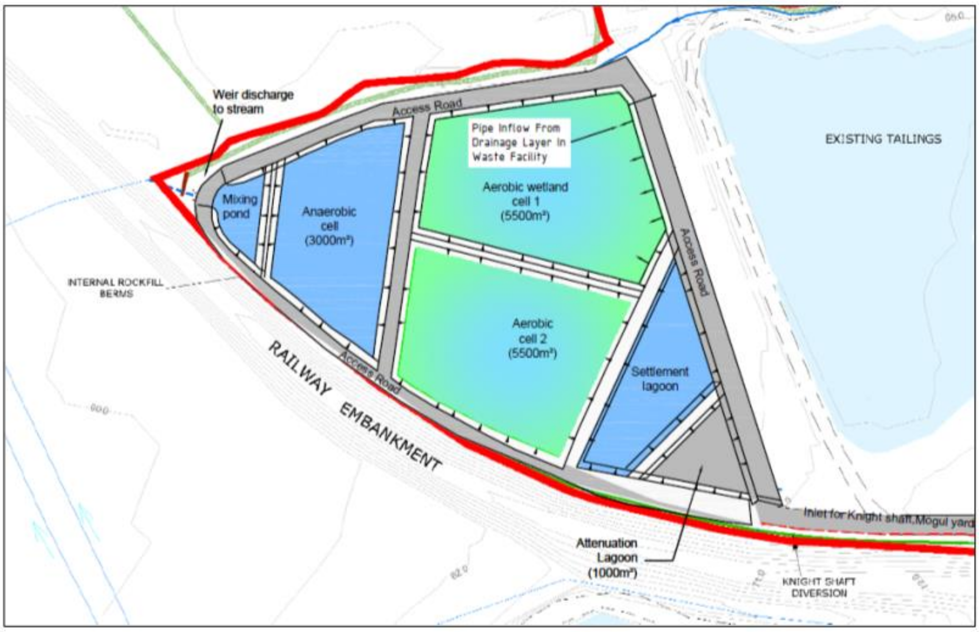


Figure 1-2
Wetland Treatment Area Layout



1.4 Site Visit and Sampling

A site visit was undertaken by an SLR field engineer on 3rd July 2017. The visit was arranged through liaison with Dr Wayne Cox, a Senior Geologist, of the Exploration and Mining Division, at the Department of Communications, Climate Action & Environment. Dr Cox provided information relating to the system on site and provided copies of reports prepared by IEPA regarding the site and impacts surrounding area. This information was used to target the site investigation.

The site walkover showed the wetland area to be heavily vegetated and overgrown. Photographs (shown in Appendix A) show the current vegetation within the aerobic, anaerobic and settlement lagoon area. A hand auger was used in each of the areas to obtain samples of the substrate. Typically the auger was advanced to 0.85m at each location and samples were taken and collected in clean sampling containers which were double bagged for protection during transport. The encountered material was described and the details are shown below:

Table 1-1
Wetland Sampling and Description

SLR Sample No.	Area	Description
39201	Aerobic Wetland Cell 1	Brown, clay rich soil
39202	Aerobic Wetland Cell 1	Brown sandy clay soil
39203	Anaerobic Cell	Grey, moist, soft clay material
39204	Anaerobic Cell	Grey, moist, soft clay material
39205	Settlement Lagoon	Dark grey, sandy clay
39206	Settlement Lagoon	Dark grey, sandy clay

1.5 Sequential Analysis

One of the issues regarding understanding the geochemistry of mine type wastes (including wetland sediments which might require removal) is that traditional wet chemistry does not distinguish the form of the metal that is detected. Consequently a more applied analytical procedure is required to understand the form of the metal or metalloid in the substrate of a wetland.

The use of sequential analysis is not new and it has been used successfully to understand the likel y partition of metal in substrates. The Tessier method[3] uses a combination of extraction techniques to classify the metals/metalloid as being:

Fraction 1. Exchangeable – Studies on sediment or their major constituents (clays, hydrated oxides of iron and manganese, humid acids) have shown the adsorption of trace metals.

Fraction 2. Bound to Carbonates – significant trace metal concentration can be associated with sediment carbonate compounds.

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[3] Tessier. A, P.G.C Campbell and M.Bisson 1979 Sequential Extraction Procedure for the Speciation of Particulate Trace Metals. Analytical Chemistry Vol.51, No.7 p844-851

Fraction 3. Bound to Iron and Manganese Hydroxide. It is well established that iron and manganese oxides exist as nodules, concretions, cement between particles, or simply as a coating on particles; these oxides are excellent scavengers for trace metals and are thermodynamically unstable under anoxic conditions (i.e. low Eh).

Fraction 4 Bound to Organic Matter – Trace metals may be bound to various forms of organic matter: living organisms, detritus, coatings on mineral particles, etc. The complexation and peptisation properties of natural organic matter (notably humid and fulvic acids) are well recognized, as is the phenomenon of bioaccumulation in certain living organisms. Under oxidizing conditions in natural waters, organic matter can be degraded, leading to a release of soluble trace metals.

Fraction 5 Residual – Once the first four fractions have been removed, the residual solid should contain mainly primary and secondary minerals, which may hold trace metals within their crystal structure. These metals are not expected to be released in solution over a reasonable time span under the conditions normally encountered in nature.

The geochemistry of the substrate will be a key parameter in understanding if the metal is in a form designated as toxic when classifying the material for disposal and/or it also will impact the efficiency of extracting the metal as a resource.

The metals/metalloids which were selected for analysis using the Tessier method are:

- Zinc
- Lead
- Arsenic; and
- Cadmium

In addition the moisture content and stone content was also established along with the Total Organic Carbon analysis. All analysis was undertaken by I2 Analytical in the UK.

1.6 Results and Interpretation

Care is needed when interpreting the results of sequential extraction experiments. The main issues with sequential extraction interpretation are that sulphides can be mobilised in acidic solutions (extracts) and therefore their presence in a material cannot be reliably represented in the results of Tessier extractions[4]. In addition, in respect of anoxic sediments, sampling and storage can impact the distribution of metal speciation which can bear little resemblance form the original speciation.[5]

The total metal analysis for each of the substrates is presented in Table 1-2, below. The complete analysis is presented as Appendix B. The results of the analysis were compared to that previously undertaken for a biochemical reactor designed to remove zinc from a mine water discharge at Nenthead, Cumbria.

The results of the sequential extraction are presented in Table 1-2, below. In summary the following observations can be made:

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[4] Peltier.E., Dahl.A.L. and Gaillard J-F. 2005. Metal Speciation in Anoxic Sediments: When Sulphides can be Construed as Oxides. *Enviro Sci. Technol vol* 39, pp311-316

[5] Forstner. U and Kersten M., 1988,. Assessment of Metal Mobility in Dredged Material and Mine Waste by Pore Water Chemistry and Solid Speciation in Chemistry and Biology of Solid Waste : Dredged Material and Mine Tailings Ed W Salomons and U Forstner. Springer – Verlag p214 – 233

Table 1-2
Results of Total Metal and General Analytics

Sample Reference		Aerobic	Anaerobic	Settlement
Stone Content	%	< 0.1	< 0.1	< 0.1
Moisture Content	%	44	48	32
Total Organic Carbon (TOC)	%	4.4	5.9	2.5
Arsenic (aqua regia extractable)	mg/kg	21	48	30
Cadmium (aqua regia extractable)	mg/kg	3.1	14	2.0
Lead (aqua regia extractable)	mg/kg	890	1900	940
Zinc (aqua regia extractable)	mg/kg	420	1500	410

1.6.1 Settlement Pond.

This structure is designed to capture the first flush of drainage from the tailings dam, capturing sediment and provide flow balancing to prevent the wetlands from being washed out. The contents of this structure would expect to comprise metals in a variety of forms (based on the tailings chemistry) and also potential encouraging oxidation reactions – such as iron hydrolysis. This structure would also be expected to trap detritus, (organic matter), potential primary sulphide minerals and also host/country rock material sediment. This would explain the observed extraction chemistry.

Arsenic is predominantly held as residual compounds and bound to iron and manganese oxides. The residual compounds are most likely related to the primary and secondary minerals in the tailings. In addition arsenic is known to adsorb strongly to iron oxide and therefore the residual concentration may be a reflection of the arsenic held in the structure of the iron oxide and not extracted.

Cadmium is bound to organic matter and the iron and manganese oxides. There may also be a degree of sulphide in the settlement pond (i.e. the oxidation which produces the extract for organic matter may also comprise some sulphide).

Lead, unlike the other metals and metalloid, has an increased proportion of carbonate in the settlement pond, most likely related to the host rock/tailings.

Zinc is predominantly bound to iron/manganese oxides and organic matter. This is similar to the other metals and metalloids, possibly existing as residual sulphides and also the presence of organic matter detritus collecting in the settlement pond.

1.6.2 Aerobic System

This system is designed to provide an oxidising environment such that iron hydroxide forms and is precipitated as an oxyhydroxide. It also can act as a natural filtration system to reduce suspended solids (which pass from the settlement lagoon).

It would be expected that the metals and metalloids are shown to be bound to iron and manganese oxides. This is the case for arsenic which also has a higher percentage bound as residual. The latter is probably because the extraction was not sufficient to liberate the strongly bound arsenic in the iron hydroxide structure. Cadmium is 100% associated with the iron hydroxide, with lead bound to the oxides and to carbonates. The latter may be a function of the pass through of the lead carbonate from the settlement pond.

Zinc has a similar percent distribution to lead. What is interesting is the increased proportion of zinc carbonate. As this was not identified in the settlement pond, it suggests that limestone was present in the aerobic system as a building material or filter medium and this has caused secondary precipitation of the zinc.

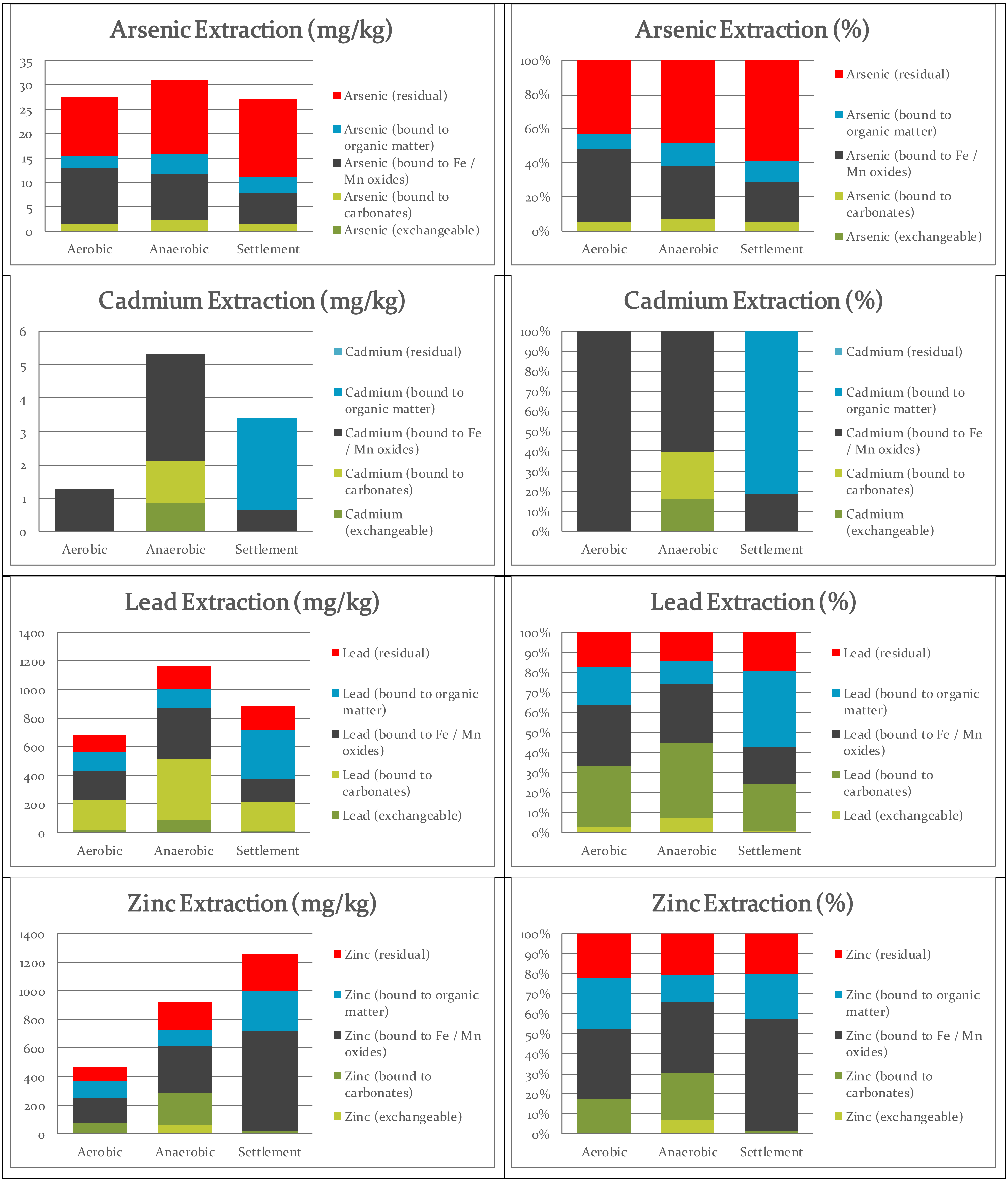
1.6.3 Anaerobic System

This system is designed to remove heavy metals as sulphides through the actions of sulphate reducing bacteria. As a by-product of the bacterial action, alkalinity is added to the water which drains through the system.

The arsenic is shown to be bound in a similar configuration to the settlement pond. The cadmium is seen to be bound to iron and manganese oxides and carbonate. It is not unusual for anaerobic systems to also include limestone to buffer the potential generation of humid and fulvic acids generated by the presence of organic matter used by the sulphate reducing bacteria. Therefore secondary cadmium carbonate appears to be present and was generated in the wetland system.

Residual iron not trapped by the aerobic system would also precipitate in an anaerobic system which explains the control on the cadmium. Lead and zinc have increased proportions of carbonate in the anaerobic system which supports the theory that limestone is likely to be present and forms secondary minerals. The extraction of organic matter bound metals may also represent a proportion of sulphide which are oxidised during the extraction.

Table 1-3
Results of Sequential Extraction



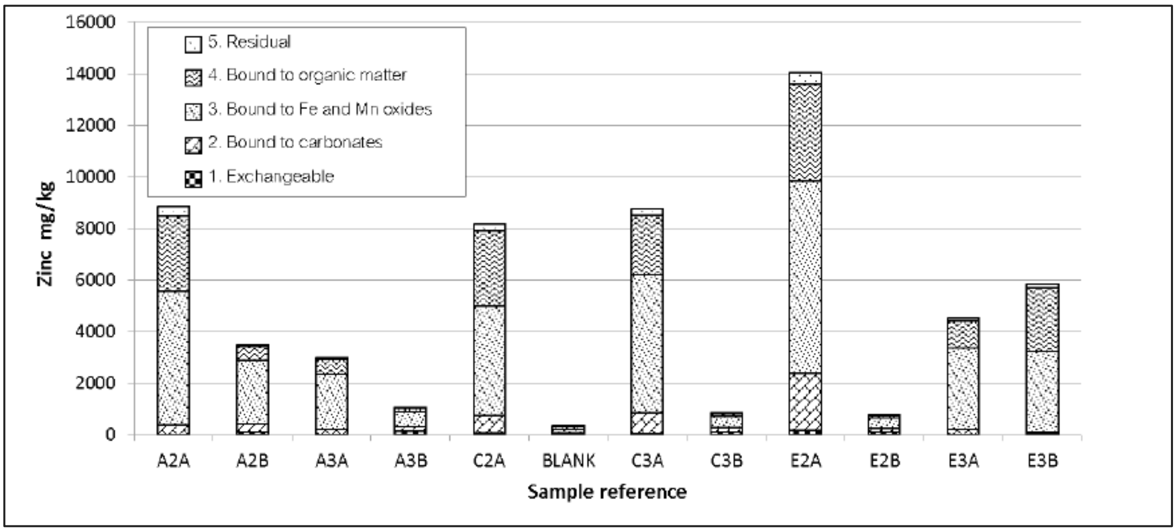
1.6.4 Nenthead Biosystems Comparison

The Nenthead reactor is an anaerobic treatment system operated by the Coal Authority in the UK. It currently treats acidic mine water with high concentrations of zinc. Consequently the results of samples taken at Nenthead have been compared with the zinc distribution in the Silvermines anaerobic wetland.

The comparison suggests that where the concentration of zinc in the Nenthead reactor is similar to that in the anaerobic system, the distribution is similar to that in the anaerobic wetland at Silvermines. For example, the largest percentage of controlling media at Nenthead was organic matter (possibly sulphide) iron/manganese oxide and carbonate. This correlates well with the results from the Silvermines anaerobic wetland. The comparison provides confidence that the extraction, and therefore the resulting interpretation are robust.

Figure 1-3

Zinc Concentrations from a Range of Sample Locations in Nenthead Bioreactor (Bailey et al 2015)[6]



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[6] Bailey M, A Jarvis and C Grundy, 2015. Characterisation of Passive Treatment System Substrates and Potential for Zinc Recovery. Presented at 10th International Conference on Acid Rock Drainage & IMWA Annual Conference

2.0 Material Classification

2.1 Waste Management and Disposal Considerations

The substrates in the wetland will have a finite life. At some future date, the depleted substrate will need to be replaced and disposed of according to the current waste legislation. Given the concentration of metals in the substrate there is a potential the material may be hazardous in terms of disposal.

In terms of measuring the hazardous components in the waste, a proprietary piece of computer software (Hazard waste Online)[7] can be used. This methodology, adapted with the software, is acceptable by the UK Environment Agency for waste classification purposes. The assessment is therefore undertaken in accordance with UK guidance WM2 (now reissued as WM3, post May 2015)[8]. Additionally the software used enables a number of waste samples to be assessed simultaneously and is pre-populated with the relevant European Risk Phrases referenced by the Paper Tool. The software also considers cumulative risk in the same way the Paper Tool suggests is applicable.

The assessment also allows more detailed assessment in relation to the actual compounds likely to be present in the waste. For example chemicals reported as elemental forms (e.g. iron, zinc or lead) are selected as specific compounds available within the software database, to represent the geochemical form most likely to be present in the waste (e.g. oxides, sulphates or chlorides). To do so however requires a conversion factor to be applied to the w/w percentage concentration of each chemical as reported in the laboratory analysis and although this could be undertaken in the Paper Tool there is no dedicated flow procedure for it.

The Paper Tool requires that a number of initial steps are undertaken. The first decision is the EWC code for the waste and whether that material is hazardous, non-hazardous or a mirror entry (potentially hazardous or non-hazardous depending on the presence of certain contaminants).

2.1.1 European Waste Catalogue Code

The treated mine water waste products are classified as ‘wastes from soil and groundwater remediation ‘or European Waste Catalogue code 19 13 as follows:

19 13 Wastes from Soil and Groundwater Remediation

Within the EWC, wastes that are hazardous are either:

- Absolute entries – hazardous waste regardless of any threshold concentrations (for example acid-generating tailings would be regarded as hazardous waste without testing);
- Mirror entries – hazardous waste only if dangerous substances are present above threshold concentrations (for example waste soil could be hazardous or non-hazardous waste depending on the concentration of contaminants present).

Regarding the remediation of mine water (a form of contaminated groundwater) in terms of the spent wetland substrate waste there are two relevant categories in the EWC as follows:

- 19 13 05 - sludges from groundwater remediation containing hazardous substances – Mirror entry
- 19 13 06 – sludges from groundwater remediation other than those mentioned in 19 13 05 – Mirror entry.

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[7] https://www.hazwasteonline.com/default.aspx

[8] EA, 2015. Guidance on the Classification and Assessment of Waste (1st Edition 2015) Technical Guidance WM3. May 2015. LIT 10121

Therefore the appropriate waste code will either be 19 13 05 or 19 13 06, dependent on its composition and hazardous property assessment.

2.1.2 Chemical Composition

The chemical composition of the treatment substrates is enriched in lead (3730mg/kg or 0.37%), zinc (2330 mg/kg or 0.23% with lesser concentrations of arsenic and cadmium.

Understanding the form of the metal is key to determine if the waste is hazardous or not. By the very nature of the wetland treatment system, the settling pond and aerobic wetland would usually have a positive redox (i.e. >100mV) with the anaerobic predominantly reducing (that is, oxidation reduction potentials less than -100 millivolts) and neutral to slightly alkaline ph. In the presence of dissolved sulphide ions (H_2S), metals can be precipitated/formed. The sequential extraction suggests the lead and zinc are likely to be bound to:

- Organic matter;
- sulphides,
- carbonates, and
- Iron oxides.

These have different toxicity phrases (where they are present) and therefore have different thresholds for hazardous classification. The Haz Waste on-line assessment is included as Appendix C.

The preliminary assessment of the material assumed that the lead and zinc were in the form of chromates which is the most toxic form and would have classified most of the wetland substrate as hazardous in terms of disposal. When the form of the metal was changed to carbonate and oxide, the results of the Haz Waste online assessment indicate that, based on the concentrations of the lead and zinc, the anaerobic material only is hazardous based on the cumulative effects of lead and zinc. The aerobic and settlement pond material is classified as non-hazardous.

This can only be viewed as a preliminary assessment as it only constitutes the assessment of one sample from each of the substrates, however it does show the benefit of understanding the form or speciation of the metal in the substrate.

3.0 Wetland Sediment Reprocessing

3.1 Potential for Resource Recovery

There have been a number of studies which examine the potential for metals to be removed economically from acidic metalliferous mine drainage.[9] In addition, these have also included presenting technical issues which need to be considered when examining the resource recovery from mine drainage treatment schemes. [10].

It has also been shown that manganese (as manganocrete) has been harvested in the US for reuse in ceramics[11] and iron oxy-hydroxide has also been marketable.[12].

One of the latest studies[13] indicates the metals removed through conventional mine drainage treatment using lime makes the metal hydroxide precipitates not amenable to economic metal recovery. This is because economic recovery requires the creation of high metal purity and specific forms (other than hydroxides) needed by the secondary source processing facilities.

The results of the analysis showed that the metal sediment also had 2.5% - 6% organic carbon content. This is not surprising, especially for the anaerobic wetland which would require organic matter to provide a growing medium for sulphate reducing bacteria. The presence of organic matter can make metal extraction more inefficient and therefore preconditioning of the substrate would be required.

3.1.1 Pre-conditioning of Wetland Substrate

There are some key considerations which will need to be addressed as part of the conditioning phase of the spent wetland substrate. This will need to consider the following:

- Certain metals and metalloids (such as As and Hg) can make the metal recovery process less economically viable. At Silvermines these may not be an issue as arsenic is relatively low in the discharge and is not elevated in the substrate – although mercury is not analysed at present.

- The primary issue regarding metal recovery is the uncertainty as to their specific chemical form within the substrate matrix. The sequential extraction indicates that lead and the zinc are held as carbonates, sulphides (probably) and adsorbed to iron oxide.

- Leaching of the metal with subsequent recovery from the solution is a proven technology, however in order for leaching to be effective the organic and limestone fractions may need to be removed by a number of processes considered below:

    - If the physical location of the metal within the substrate column is known, visual sorting may be used to reduce the overall volume of material requiring subsequent treatment. Basic dewatering such as air drying could reduce the energy costs associated with roasting, and/or aid in the efficiency for the limestone material separation.

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[9] MEND Treatment Committee. 1991. Study on Metals Recovery/Recycling From Acid Mine Drainage. Phase 1A: Literature Survey

[10] Gusek JJ, T R Wildman and K.W Conroy. 2006. Conceptual Methods for Recovering Metal Resources from Passive Treatment Systems. Paper presented at the 7th International Conference on Acid Rock Drainage (ICARD), March 26-30, 2006, St. Louis MO. R.I. Barnhisel (ed.) Published by the American Society of Mining and Reclamation (ASMR), 3134 Montavesta Road, Lexington, KY 40502

[11] Denholm, Clifford, Timothy Danehy, Shaun Busler, Robert Dolence, and Margaret Dunn. 2008. Sustainable Passive Treatment of Mine Drainage: Demonstration of Manganese Resource Recovery. Paper was presented at the 2008 National Meeting of the American Society of Mining and Reclamation, Richmond, VA. June 14-19, 2008. RI Barnhisel (Ed) Published by ASMR, 3134 Montavesta Rd, Lexington, KY 40502

[12] Hedin R.S. 2002. Recovery of marketable iron oxide from mine drainage, presented at the 2002 West Virginia Surface Drainage Task Force Symposium, April 16-17, Moragntown, WV.

[13] Figueroa L and C. Wolkersdorf. 2014. Electrochemical Recovery of Metals in Mining Influenced Water: State of the Art. In An Interdisciplinary Response to Mine Water Challenges – Sui, Sun & Wang (eds). China University of Mining and Technology Press, Xuzhou.

- ○ Following basic treatment, roasting and/or composting could be used to separate the organic material. Additional oxidation such as chlorination may also be required to leach remaining refractory organic compounds which might affect the leaching process.
- ○ Screening or gravity separation could be potential processes for separation of the limestone fraction. Air-drying of the substrate may provide for better separation if composting is used for organic material removal. If roasting is used for organic removal, screening will be even simpler as the limestone will easily separate from the dry ash and solid residue.
- ○ Following removal and separation of the organic and limestone fractions, actual leaching of zinc could proceed. The pregnant solution could then be processed through conventional adsorption processes, such as activated carbon or ion exchange, for final metals recovery.
- ○ One aspect of the metals recovery process described above that must be evaluated is the potential acceptability of pre-treated materials at existing recovery facilities. Pre-treatment of the BCR substrate for separation of organic material and limestone may produce a material that could be shipped directly to an existing facility. This could have a significant positive impact on the overall economic viability of metals recovery from SRBR substrates. It would however need a processing facility close to the proposed mine water treatment scheme.

3.1.2 Metal Extraction

There has been limited research into the field of metal processing from wetland treatment systems. That which has been published recently [14] indicates that there are two main aspects which needed to be considered in assessing the suitability of a substrate to be reprocessed:

1. Is the metal of sufficient grade to make recovery economic?; and
2. Which current or future technologies are most appropriate for metal reserves produced by engineered wetland treatment systems?

Discussions with professionals who operate in the mining field [15] indicate that there are optimum concentrations of metal within an ore which determine if the metal can be mined. The Environment Agency (2012) terms this the 'cut-off value' and it is specific to the metal and ore being mined. It is indicated that concentrations of lead or zinc would need to be in excess of c.1% (percent w/w) or 10,000mg/kg to make extraction of the metal economically viable.

Although a full sampling and analytical suite of the different treatment zones in the wetland is beyond the scope of the study, it is likely that the metals are low-grade and dispersed as fine-grained particulates in the wetland substrate (as precipitates or adsorbed). Therefore this presents the metals in a form which are not typical of metal extraction from high grade ores.

1. Thermal extraction of metal. This technique is usually undertaken to extract metals from relatively high grade ores of metals such as copper, zinc and lead. Consequently the application of thermal techniques may not be fully suited to the metals in the wetland substrate.
2. Chemical leaching of the metal.
3. Biological leaching of the metals. This typically involves using techniques such as bioleaching and/or soil washing.

Traditional leaching of metals yielded results which were little more effective than thermal treatment in terms of the quantity of metal required in the material being leached. However bioleaching both or metal recovery and for remediation of metal contaminated sediments were able to extract metals at lower concentrations.

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[14] Environment Agency (2012). Mitigation of pollution from abandoned mines. Part 2: Review of resource recovery options from the passive remediation of metal-rich mine waters. Ref: SC090024/R2

[15] Jim Gusek Sovereign Consultants, Denver (pers comm.), 2017

Bioleaching usually takes the form of three different biologically mediated extractions. This occurs directly by bacterially mediated oxidation of metal sulphides using zinc sulphide as an example):

$ZnS + 2O_2 \rightarrow ZnSO_{4\ (aq)}$ Eq 1

Or indirectly where metal sulphide oxidation and dissolution occurs as a result of reactions with ferric iron in acidic conditions:

$ZnS + 2Fe^{3+} \rightarrow Zn^{2+}_{(aq)} + Fe^{2+} + S_0$ Eq.2

Where the metal is held in a non-sulphide form the organic acid generated by bacteria and fungi dissolve or complex the metal ions (example below is zinc oxide and citric acid):

$ZnO + C3H5O(COO)_3 + 2H+ \rightarrow (C3H5O(COO)Zn)^- + H_2O$ Eq.3

A compilation of the information provided in Environment Agency (2012), coupled with information from discussions in industry, and has enabled the following summary table to be prepared regarding some examples of leaching, soil washing and bioleaching trials.

Table 3-1
Examples of Leaching Studies on Metals from Substrates (after Environment Agency, 2012)

Outline of study	Metal Concertation in Substrate	Recovery/Removal	Reference
Solvent extraction of waste rock recycled acidic heap leach. Followed by solvent extraction, neutralisation and Zn electro winning	c.15% Zn	Not reported	Peterson and Dixon (2007)[16]
Bioleaching of steel waste using Fe-oxidising bacterium	c.1%	Not reported	Bayat et al (2009)[17]
Soil leaching low grade ore in the presence of fungi and agricultural waste pre-soaked in sulphuric acid generating organic acids.	7245 mg/kg Cu 201 mg/kg Zn	68% Cu 46% Zn	Mulligan et al (2004)[18]
Soil washing with EDTA, acid mixing, addition of ferric sulphate with	1730 mg/kg Cu	60% Cu	Di Palma and Medici (2002)[19]

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[16] Petersen J and Dixon D.G., 2007. Modelling of zinc heap bioleaching. *Hydrometallurgy* 85 127-143.

[17] Bayat,O.,Sever,E.,Bayat,B.,Arslan,V. and Poole,C.,2009.Bioleachingof zinc and iron from steel plant waste using Acidithiobacillus ferroxidans. *Applied biochemistry and Biotechnology* 152, 117-126

[18] Mulligan,C.N.,Kamali,M. and Gibbs,B.F.,2004. Bioleaching of heavy metals from low grade mining ore using *Aspergillus niger*. *Journal of Hazardous Materials* 110, 77-84

[19] Di Palm, L. and Medici,F.,2002. Recovery of copper from contaminated soil by flushing. *Waste Management* 22, 883-886

Outline of study	Metal Concertation in Substrate	Recovery/Removal	Reference
addition of Ca and Na hydroxide.			
Soil decontamination using chelating agent DTPA.	1410 mg/kg Zn 12000 mg/kg Pb 675 mg/kg Cu	74% Zn 100% Pb 55% Cu	Hong et al (2002)[20]
Bioleaching contaminated sediment including conditioning and addition of 2% elemental sulphur.	1958 mg/kg Zn 135mg/kg Pb 9 mg/kg Cd	61-81% Zn Pb immobile 61-81% Cd	Seidal et al (2004)[21]
Bioleaching metal plating sludge.	22900-22980 mg/kg Zn 828-850 mg/kg Pb 325-341 mg/kg Cd	97% Zn 67% Cd 84% Pb	Bayat and Sari (2010)[22]

3.1.3 Advances in Biomine Research

Bio mining is a technique of extracting metals from ores and other solid materials which involves the use of bacteria or fungi. These organisms secrete different organic compounds that chelate metals from the environment. This process is described in Eq.1-3 above.

One of the key areas of European research efforts in this area is biohyrometallurgy. This has recently been explored through the BIOMINE (Biotechnology for Metal bearing materials in Europe) research project funded through the European Union under CORDIS (Community Research and Development Information Service).[23]

The BIOMINE project was designed to assess the potential for optimising traditional bio-hydrometallurgy and to enhance the biotechnological processes for recovery of metals. The BIOMINE project brief considered the extraction of metals from primary materials (such as ores and concentrates) and secondary materials (such as mining wastes, metallurgical slags, and combustion / power plant ashes). However it does not state if recycling spent wetland substrates is being or has been considered as part of the project objectives.

The biotechnologies investigated, have included bioleaching, bio-oxidation, bio-sorption, bio-reduction, bio-accumulation, bio-precipitation, bio-flotation, bio-flocculation, and bio-sensors.

The main objective has been to determine if smaller scale Biosystems can be used as an alternative to smelting and roasting to extract metals from different substrates. The results of the study as reported in the BIOMINE summary report concluded the following:

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[20] Hong,A.,Chelsea,L.,Banerji,S.K., and Wang,Y.,2002. Feasibility of metal recovery from soil using DTPA and its bio stability. *Journal of Hazardous Materials*, B94, 253-272.

[21] Seidel,H.,Loser,C.,Zehnsdorf, A.,Hoffmann,P and Schmerold,R.,2004. Bioremediation process for sediments contaminated with heavy metals: feasibility study on a pilot scale. *Environmental Science and Technology*,38 1582-1588.

[22] Bayat,B and Sari,B.,2010. Comparative evaluation of microbial and chemical leaching processes of heavy metal removal from dewatered metal plating sludge. Journal of Hazardous Materials, 174, 763-769.

[23] http://cordis.europa.eu/result/rcn/49662_en.html

Bio hydrometallurgical treatment can only be successful if this led to the following when compared to conventional extraction techniques:

- substantial improvements for metal production by increased recovery,
- reduced costs,
- reduced energy demands,
- increased revenue,
- access to new resources,
- Better compliance with the environmental regulations and/or better acceptance from the public of the mining activities in Europe.'

The results of the project indicate that mining operators should consider bio hydrometallurgy for the viable processing of metal-bearing minerals. This study suggests that commercially available bio metallurgy for the extraction of low grade metals (such as in a spent wetland substrate) is developing, although it does show significant opportunities from a viable resource perspective.

A review of some recent research (e.g. Scippers et al, 2014)[24] indicate that up to now, bio mining has merely been used as a procedure in the processing of sulphide ores and uranium ore, but laboratory and pilot procedures already exist for the processing of silicate and oxide ores (e.g., laterites), for leaching of processing residues or mine waste dumps (mine tailings), as well as for the extraction of metals from industrial residues and waste (recycling).

Therefore it appears there is viability for the process to be examined at a commercial level, however at this stage, it is not known if there is any such study being undertaken in the UK.[25]

3.1.4 Metal Specific Recovery: Zinc

A review of the literature has indicated that one of the most likely metals which could be exploited successfully from spent wetland substrate material is zinc. Whilst the extraction efficiency will be reliant upon the type of substrate used and the other metals and metalloids present, the potential for zinc extraction has been considered further below.

Discussions with Hugh Potter of the Environment Agency revealed that a PhD thesis was completed in 2016 at University of Newcastle (Bailey, 2016)[26] which examined the extraction of metals from two wetland substrates in Nenthead (pilot systems) and Force Crag (full scale system), in Cumbria UK. The latter are two down-flow compost bioreactors, treating zinc-rich discharges. The Nenthead system alone was examined in terms of metal extraction from the substrate, a summary of the findings include:

- The Nenthead substrate contained 7,900mg/kg zinc in an upper substrate and 2,300mg/kg zinc in a lower horizon, after two years of operation.
  - Complete recovery of zinc through acid leaching was observed after c.100 hours across a range of acid leach testing (0.02M, 0.1M and 0.5M sulphuric acid)
  - Extraction efficiencies for lead and copper were comparatively poor reaching 68% and 58% respectively.
  - Biological leaching took 23-37 days to obtain an equivalent result.
  - On the basis of the most readily extractable fractions in the substrate (using the Tessier method), the metals investigated were ordered in the following sequence:

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[24] Scippers A., Hedrich S., Vasters J., Drobe M., Sand W., and Willscher S. 2014. Bio mining : metal recovery from ores with microorganisms. Adv Biochem Eng Biotechnol 141 pp1-47.

[25] Personal Communication – Barrie Johnson – 2017.

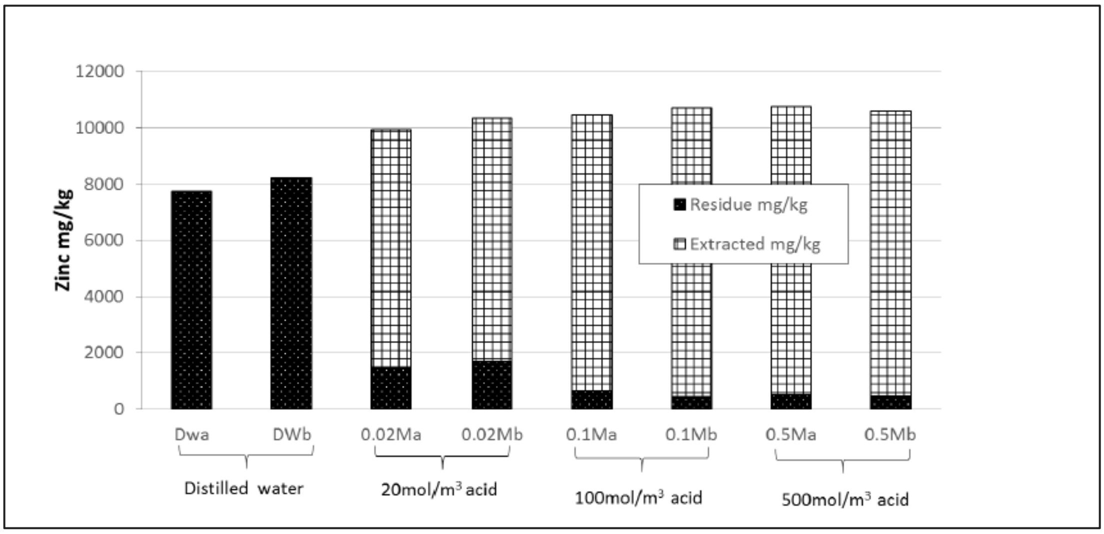
[26] Bailey, M.T. July 2016. Recovering Resources from Abandoned Metal Mine Waters: An Assessment of the potential options at passive treatment systems. Unpublished PhD Thesis, School of Civil Engineering and Geosciences University of Newcastle, UK.

Mn > Zn > Ni> Cd> Fe> Pb> Cu

- The findings suggest that sieving may offer a means of concentrating the zinc rich fraction of the substrate.
- Whilst bioleaching took longer, it also provided a low iron extraction whilst still extracting 100% zinc and 80% nickel and cadmium. This may be preferable when considering the potential issues of excess iron in relation to the process engineering associated with a full scale extraction system.

The results of the zinc acid extraction work are summarised below

Figure 3-1
Zinc Concentration from Leachate and Residue after Acid leaching (Bailey et al 2015)[27]



There have been limited practical demonstrations or pilot studies which have examined the recovery of heavy metals from within spent wetland substrate material, although there have been a number of studies examining the potential for zinc reuse/recovery.[28] [29] [30] [31]. More recent studies have also examined various influences[32] on the economic viability of zinc recovery. These have identified other potential opportunities:

- Zinc metal production. Higher grades/concentrations of zinc in the source are considered the most suitable (this will require preconditioning of the spent wetland substrate); and
- Micronutrient Fertilizers production. Where the zinc can be supplied in various forms although it is preferred to be more soluble with limited or no co-metals/contaminants.

The viability of the spent wetland substrate reuse would depend on the likely feed quality and how much pre-conditioning it might require. The required zinc smelter feed quality (based on US examples) are shown below:

---

[27] Bailey M, A Jarvis and C Grudny, 2015. Characterisation of Passive Treatment System Substrates and Potential for Zinc Recovery. Presented at 10th International Conference on Acid Rock Drainage & IMWA Annual Conference

[28] Zinck J.. Disposal, Reprocessing and Reuse options for Acidic Drainage Treatment Sludge. Poster presentation at 7 th International Conference on Acid Rock Drainage March 26-30 2006. St Louis, USA.

[29] Zinck J 2005. Review of Disposal, Reprocessing and Reuse Options for Acidic Drainage Treatment Sludge. Mine Environment Neutral Drainage Proram, Canada.

[30] Luptakova A, S Ubaldini, P Fornan and E Macingova 2012. Physical-chemical and Biological- chemical methods for Treatment of Acid Mine Drainage. *Italian Association of Chemical Engineering Vol 28 p115 120*

[31] Olper M and M Maccagni. 2008. From C.Z.O to Zinc Cathode Without Any Pre-treatment. The EZINEX Process. The Southern African Institute of Mining and Metallurgy. Lead and Zinc. pp85-97

[32] Whysner K, L Figueroa, E Petri and M Holmes 2012. Economic Recovery of Zinc from Mining Influenced Water (MIW). USEPA Hardrock Mining Conference.

Table 3-2
Permissible Zinc Smelter Feed Concentration

Constituent	Percent
Moisture	< 18% water
Zinc	>57%
Sulphur	<38%
Other	<5%
Dry weight % unless shown	

For fertilizer production the requirements are plant specific and often negotiable but for comparative purposes the following is considered representative:

Table 3-3
Fertiliser Sourcing Specifications

Component	Concentration
Zinc	>30%
Aluminium and iron	Flexible but low (Fe <1% and Al < 0.5%)
Cadmium and lead	Cd < 2% and Pb < 5%
Calcium and Magnesium	< 2%

Typically, the inorganic form of zinc in fertiliser has been identified and these are summarised in Table 3-4 below[33].

Table 3-4
Inorganic Zinc Present in Fertiliser

Name	Formula	Content (%)
Zinc sulphate monohydrate	$ZnSO_4$. H2O	36
Zinc sulphate heptahydrate	$ZnSO_4$.7H2O	22
Zinc oxysulphate	$xZnSO_4.x7H_2O$	20-50
Basic Zinc Sulphate	$ZnSO_4.4Zn(OH)_2$	55
Zinc oxide*	ZnO	50-80
Zinc carbonate	$ZnCO_3$	50-56
Zinc chloride	$ZnCl_2$	50

---

[33] Alloway B J.2008. Zinc in Soils and Crop Nutrition. International Fertilizer Industry Association and International Zinc Association, Brussels, Belgium and Paris, France. p84-86

Name	Formula	Content (%)
Zinc nitrate	$Zn(NO_3)_2.3H_2O$	23
Zinc phosphate	$Zn_3(PO_4)_2$	50
Zinc frits	Fritted glass	10-30
Ammoniated zinc sulphate solution	$Zn(NH_3)SO_4$	10

*Insoluble

A number of these compounds could theoretically either be present within the spent substrate or could be developed by using the zinc from the treatment substrate with processing.[34] The sequential analysis indicated that zinc carbonate and probably oxide are most likely to be present in the substrate.

The above examples are based on US requirements and not the EU and these will need to be researched further. The sampling and analysis to date is only preliminary at this stage and has been designed to prove a principle regarding the reprocessing of wetland substrates. However if the sampled wetland metal concertation is considered typical, some comparisons can be made. There is no dry weight analysis for calcium, magnesium, aluminium or iron for the wetland matrix material. It is also appreciated that pre-conditioning of the substrate would be required to remove organic matter and limestone. Notwithstanding these restrictions, the metal analytical results for samples analysed from the reactor are compared with the input requirements for smelting and fertilizer production below.

Table 3-5
Wetland Composition Compared to Feed Concentrations

	Zn (%)	Cd (%)	Pb (%)	Moisture (%)
Wetland Samples	0.23	0.002	0.4	41
Smelter	57	< 5		< 18
Fertilizer	30	< 2	< 5	

Based on this comparison the bioreactor does not contain sufficient zinc to make smelting or fertilizer production and the moisture content is also too high. In addition the 1% ore grade cut off suggested by the Environment Agency, also indicates that there is insufficient zinc in the substrate to make its extraction through smelting or use in a fertilizer economically attractive.

However, the research has shown that extraction using leaching and bioleaching has been proven successfully at laboratory scale testing. It is also clear that the 1% cut off of zinc may not be prohibitive in terms of the extraction viability.

---

[34] Gusek, JJ, T. Wildeman and K. Conroy. 2006. Conceptual Methods for Recovering Metal Resources from Passive Treatment Systems. 2006 International Conference on Acid Rock Drainage (ICARD) and the Annual Meeting of the American Society for Mining and Reclamation, March. St. Louis.

4.0 External Communication

4.1 Points of Contact

As part of the study a number of key individuals were approached in academia, government and industry to obtain as wide a view as possible regarding the objectives of the study. The people approached are shown below, the information they provided have been incorporated into the main body of the report where relevant.

Table 4-1
Individuals Contacted During the Study

Name	Organisation	Response	Incorporated in Report
Dr Caroline Gauchotte-Lindsay	Lecturer in Environmental Engineering Division of Infrastructure and University of Glasgow	Yes	General background on current research and providing leads into phytomining[35]
Professor Paul Younger	Retired Rankine Chair of Engineering, Professor of Energy Engineering, University of Glasgow	No	Paul has retired due to ill health and was not able to provide a response
Professor. Robert M Kalin	Professor of Environmental Engineering for Sustainability Strathclyde University, Glasgow.	Yes	General comments regarding aerobic and anaerobic metal mobilisation.
Rory Doherty	Lecturer in Environmental Engineering, Queens University Belfast, Belfast.	Yes	Information on redox bioelectrical transformation and geobattery projects
Dr Mark Bailey	PhD author Newcastle University.	Yes	Copy of PhD thesis and papers presented in regard to extraction of metals from biochemical reactor substrates.
Hugh Potter	Technical Specialist (Pollution from abandoned mines) at the Environment Agency, Newcastle	Yes	Provided up to date contact details for most recent research in the UK in this area.
Dr Russell Thomas	Visiting Professor at the University of Strathclyde, Glasgow	Yes	General background and possible contacts in the field.
James Gusek	Sovereign Consultants, Denver, US.	Yes	Papers regarding the extraction of metal from biochemical reactor substrate.

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[35] http://www.nancy.inra.fr/en/All-the-news/Using-plants-to-micro-mine-metals

Name	Organisation	Response	Incorporated in Report
Dr Wayne Cox	Senior Geologist, Exploration and Mining Division, Department of Communication, Climate Action and Environment, Dublin.	Yes	Provided useful information and reports regarding Silvermines site.
Dr Robert Baker	Assistant. Professor. in Inorganic and Materials Chemistry, School of Chemistry, Trinity College, Dublin,	Yes	Did get some funding from the EPA to look at precious metal extraction from e-waste so we didn't look at lead. However from previous work we did investigate lead and tin extractions using a variety of extractants we designed and had ca 80 % efficiency for lead. This was not using scCO2 rather solvent extractions; on a large scale this may not be cost effective. The principle however does and would work, however it's a question of scale.
Dr Billy Fitzgerald	Sligo University, Head of Environmental Science (retired), Ireland.	No	Dr Fitzgerald has retired as Head of Environmental Science.
Professor David Barrie Johnson	Professor of Environmental Biotechnology, Bangor University.	Yes	Provided the following comment 'I am not aware of similar research re-commercial-scale operations, being carried out in the UK'
Environment Section	Tipperary County Council	No	Attempted to make contact with Tipperary County Council to make them aware of the project and elicit comments, but no response was received after two efforts at communication.
Pat Ryan	Landowner	Yes	The Ryan family was happy to allow access to their land for the purposes of entering the site and taking samples. Very accommodating.

5.0 Conclusions and Recommendations

5.1 Conclusions

Preliminary assessment of the wetland substrate at Silvermines has been undertaken by using sequential extraction techniques to establish the form of the zinc, lead, cadmium and arsenic in different areas of the wetland treatment system.

The results of the assessment have indicated the following:

- The engineered wetland treatment system at Silvermines appears to have trapped lead at zinc at relatively elevated concentrations.
- The metals and metalloids (arsenic) are bound predominantly to iron oxides, organic matter (sulphides) and carbonates.
- The concentrations are not likely to be sufficient for extraction of metal through smelting, although the use of bioleaching and leaching has been successfully applied in other cases.

A preliminary hazardous waste assessment using Haz Waste on line indicates the lead and zinc in the anaerobic system substrate is probably hazardous in terms of off-site disposal.

5.2 Recommendations

The sampling exercise indicates that the Silvermines wetland do appear to be overgrown and relatively full. It is likely that the wetland requires rejuvenating, and the spent wetland substrate will require disposal and / or reprocessing.

The research has shown that even with a simple preliminary sampling programme, metals are present in the substrate and therefore it is logical to assume that with a more intensive sampling strategy the distribution/extent and concentration of the metals could be realised.

Therefore it is recommended that a more complete investigation of the substrate is undertaken.

The research undertaken has also indicated that bioleaching and leaching may be viable options for extracting the metal from the wetland substrate and it is recommended that treatability/feasibility trials are undertaken to test the viability of this option.

Discussions with Sligo IT have indicated that a methodology for leaching metal is established and, subject to funding, this could be reinvigorated to establish if the metals can be successfully extracted for reuse as a resource.

There are likely to be funding mechanisms through research grants and potentially through industry and it is recommended that relevant industrial bodies are approached to discussed if reprocessing of the wetland substrate presents a prospective industry for which research funds are available.

APPENDIX A

Site Photographs

Aerobic Wetland Cell 1 Looking North

Aerobic Wetland Cell Looking South

Anaerobic Cell Looking North.

Anaerobic Wetland Cell Looking South

Settlement Lagoon Looking North East

Settlement Lagoon Looking South West

Notes:

SLR

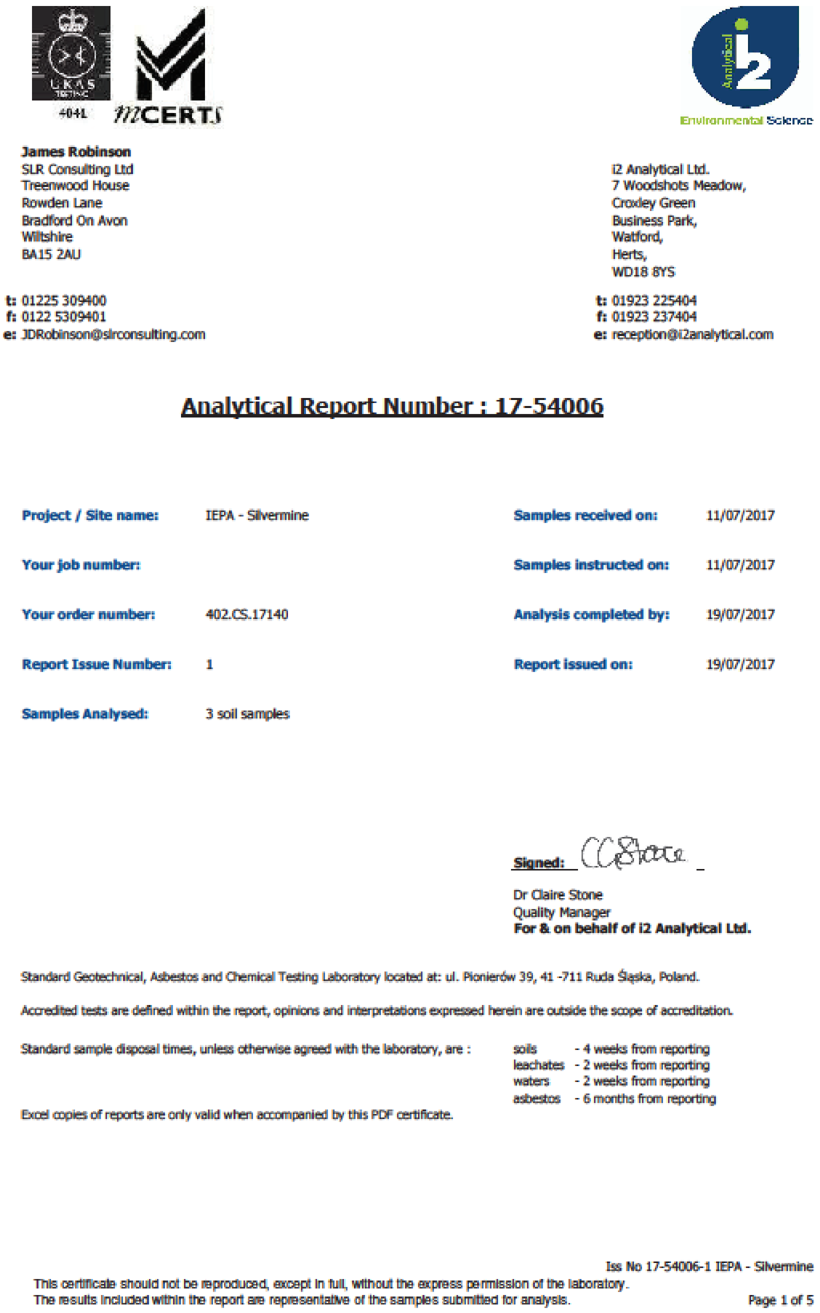
402.04857.00002

Site: WETLAND TREATMENT AREA, SILVERMINES
Project: PRELIMINARY WETLAND REPROCESSING ASSESSMENT
Date: JULY 2017
Drawing: **OBSERVATION PHOTOGRAPHS**

Appendix A

APPENDIX B

Results of Chemical Testing



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Analytical Report Number : 17-54006

Project / Site name: IEPA - Silvermine
Your job number:
Your order number: 402.CS.17140
Report Issue Number: 1
Samples Analysed: 3 soil samples

Samples received on: 11/07/2017
Samples instructed on: 11/07/2017
Analysis completed by: 19/07/2017
Report issued on: 19/07/2017

Signed: CCStone

Dr Claire Stone
Quality Manager
For & on behalf of i2 Analytical Ltd.

Standard Geotechnical, Asbestos and Chemical Testing Laboratory located at: ul. Pionierów 39, 41 -711 Ruda Śląska, Poland.

Accredited tests are defined within the report, opinions and interpretations expressed herein are outside the scope of accreditation.

Standard sample disposal times, unless otherwise agreed with the laboratory, are :

soils	- 4 weeks from reporting
leachates	- 2 weeks from reporting
waters	- 2 weeks from reporting
asbestos	- 6 months from reporting

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The results included within the report are representative of the samples submitted for analysis.

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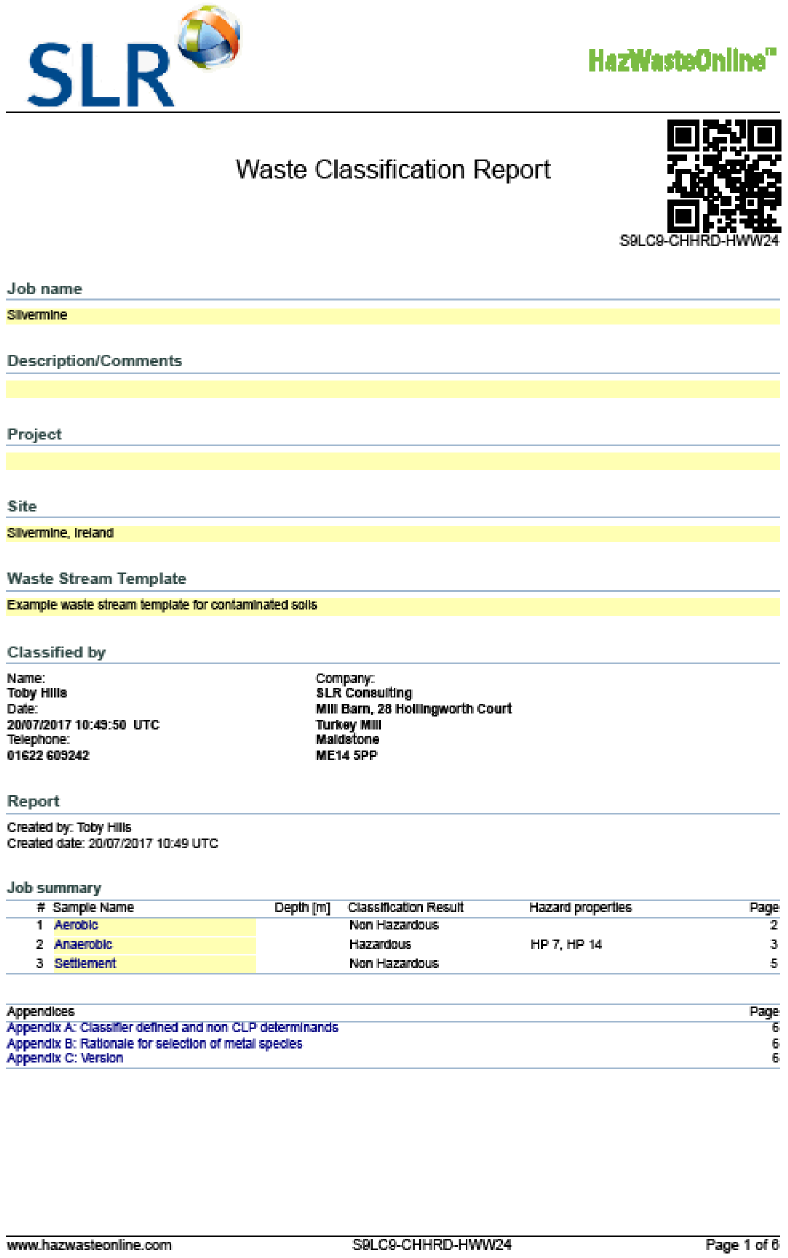
APPENDIX C

Haz Waste Online Assessment



Waste Classification Report

S9LC9-CHHRD-HWW24

Job name

Silvermine

Description/Comments

Project

Site

Silvermine, Ireland

Waste Stream Template

Example waste stream template for contaminated soils

Classified by

Name:
Toby Hills
Date:
20/07/2017 10:49:50 UTC
Telephone:
01622 609242

Company:
SLR Consulting
Mill Barn, 28 Hollingworth Court
Turkey Mill
Maidstone
ME14 5PP

Report

Created by: Toby Hills
Created date: 20/07/2017 10:49 UTC

Job summary

#	Sample Name	Depth [m]	Classification Result	Hazard properties	Page
1	Aerobic		Non Hazardous		2
2	Anaerobic		Hazardous	HP 7, HP 14	3
3	Settlement		Non Hazardous		5

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