

Guidebook for the REGENERATIS methodology

REGENERATIon of Past Metallurgical Sites and Deposits through innovative circularity for raw materials





Copyright © 2023 NWE-REGENERATIS Project partners, all rights reserved.







Authors:

Carmen Alamar (Cranfield University) Pascal Beese-Vasbender (BAV) Julien Berny (IXSANE) Andrew Buchanan (MPI) Michael Capstick (MPI) David Caterina (University of Liège) Frederic Coulon (Cranfield University) Lise Debruyne (TEAM2) Tristan Debuigne (IXSANE) Renaud De Rijdt (ATRASOL) Marc Dumont (University of Liège) Ugo Falcinelli (DUFERCO) Antoine Garandeau (TEAM2) Alina Ghinet (JUNIA) Jean-Christophe Gourry (BRGM) Stuart Higson (MPI) Victoria Huntington (Cranfield University) Cuinera Isenborghs (OVAM) Itzel Isunza Manrique (University of Liège) Adeline Janus (IXSANE) Ipek Tezyapar Kara (Cranfield University) Pauline Kessouri (BRGM) Mohamed Krid (IXSANE) Thomas Kuklok (TH Köln) Laura Lamair (SPAQuE) Laurie Lommel (ATRASOL) Niall Marsay (Cranfield University) Antoine Masse (CTP) Benoît Mignon (CTP) Sébastien Moreaux (ATRASOL) Claudia Neculau (SPAQuE) Frédéric Nguyen (University of Liège) Marta Popova (SPAQuE) Jérémie Renault (TEAM2) Alan Scholes (MPI) Tutu Sebastian (MPI) Stuart Wagland (Cranfield University) Christophe Waterlot (JUNIA) Eddy Wille (OVAM) Christian Wolf (TH Köln)

This guide is the result of teamwork among the NWE-REGENERATIS project partners.



Infographic of contents

Overview of the topics covered in this guidebook for the REGENERATIS methodology





TABLE OF CONTENTS

1		Obje	ective	es of NWE-REGENERATIS for Innovative Resource and Materials Efficiency	.5
	1.	1	Turr	ning Potential Contaminated Sites into Opportunities	.5
2		REG	ENEF	ATIS Methodology for Innovative Circularity to Recover Raw Materials (REMICRRAM)	.7
	2.	1	Intro	oduction	.7
	2.	2	REM	IICRRAM Methodology	. 8
		2.2.	1	Phase 1 – SMART PHOENIX	.9
		2.2.2	2	Phase 2 – NWE-SMARTX	.9
		2.2.3	3	Phase 3 – Business Case	10
	2.	3	Met	allurgical Sites Inventory Structure for Polluted Sites	10
3		Site	Inve	stigation - Application of Non-Intrusive Methods to Characterise Past Metallurgical Sites	11
	3. M	1 odel	Con (RAP	tribution of Geophysics to the Development of a Raw materials and Pollution Distribution	วท 11
	3.	2	Prin	ciples on how to use Geophysics	12
	3.	3	Geo	physical Survey	14
	3.	4	Buil	ding of a Raw Materials and Pollution Distribution Model (RAPIDM)	18
	3.	5	Take	e Home Message	20
	3.	6	Refe	rences	20
4		Sma	art De	cision Support Tool (NWE-SMARTX)	21
	4.	1	Desi	gn of the Smart Decision Support Tool NWE-SMARTX	21
		4.1.	1	Why and How Artificial Intelligence can be used for PMSD remediation	21
		4.1.2	2	Ambition of NWE-SMARTX	22
		4.1.3	3	NWE-SMARTX design process	23
	4.	2	NW	E-SMARTX performance	25
	4.	3	How	to use NWE-SMARTX	27
5		Reco	omm	endations for Resource Recovery from PMSD	29
	5.	1	Barr	iers	29
		5.1.	1	Legal complexity	29
		5.1.2	2	Lack of data	29
		5.1.3	3	Short-term vision	30
		5.1.4	4	Financial infeasibility	30
	5.	2	Criti	cal Success Factors in PMSD Redevelopment	31
	5.	3	Reco	ommendations	32
6		Valc	orisat	ion Options	34

Guidebook for the REGENERATIS methodology Table of contents



	6.1	Med	hanical recovery techniques	35
	6.2	The	rmal recovery techniques	35
	6.3	Hyd	rometallurgy	
	6.3.	1	Chemical leaching	
	6.3.2	2	Bioleaching	
	6.4	Eco	catalyst production	41
	6.5	Refe	erences	
7	Case	e Stu	dies - Past Metallurgical Sites and Deposits	45
	7.1	Cas	e Study 1 – DUFERCO Site at La Louvière (Wallonia)	45
	7.1.	1	Overview of historical study	45
	7.1.2	2	Scanning of the site's revalorization potential: SMART PHOENIX	47
	7.1.3	3	Site screening	47
	7.1.4	4	Design and results of geophysical investigations and lab tests	50
	7.1.	5	Pilot test design and results	51
	7.1.0	6	Output of DST's: NWE-SMARTX	54
	7.1.	7	Simulation for revalorization potential: Cost-benefit analysis	57
	7.1.8	8	Conclusion and recommendations	59
	7.2	Cas	e Study 2 – Pompey (France)	60
	7.3	Cas	e Study 3 – Teesside (United Kingdom)	63
	7.3.	1	Historical study	63
	7.3.2	2	Identification of test sites	64
	7.3.3	3	Sampling	65
	7.3.4	4	Geophysical survey	66
	7.3.	5	Laboratory tests and analysis on valorisation options	68
Sı	ummar	y		72
C	ontact l	Perso	ons	73
	Fundir	וg		74

Guidebook for the REGENERATIS methodology List of abbreviations



List of abbreviations

- AI artificial intelligence
- CEC cation exchange capacity
- EMI electromagnetic induction
- ERT electrical resistivity tomography
- GPR ground penetration radar
- GPS global positioning system
- GRA gravimetry
- HDPE high density polyethylene
- HVSRN horizontal to vertical spectral ratio of noise
- IP induced polarization
- JRC The Joint Research Centre European Commission
- KNN K-Nearest Neighbours
- MAG magnetometry
- MASW multi-channel analysis of surface waves
- MBM metal-bearing-material
- NWE-MESIS metallurgical sites inventory structure
- NWE-SMARTX smart decision support tool based on AI algorithms
- OM organic matter
- PMSD past metallurgical site and deposit
- PPPP public-private-people partnerships
- RAPIDM raw material and pollution distribution model

REMICRRAM - REGENERATIS Methodology for Innovative Circularity to Recover RAw Materials from PMSD

- SMART PHOENIX screening decision tool
- SIP spectral induced polarization
- SP spontaneous potential
- SRT seismic refraction tomography
- SVM Support Vector Machines



Prologue

Throughout history, North-West Europe has been a cradle of industrialisation, witnessing the rise of numerous metallurgical sites and deposits. These sites, scattered across the region, were instrumental in the extraction and processing of metals that fuelled the growth of industries and shaped the landscape. From the smoky chimneys of the Ruhr Valley to the historic foundries of Wallonia, these sites bear witness to the region's rich industrial heritage.

As we delve into the past, it becomes evident that the significance of these metallurgical sites extends far beyond their historical value. In the context of urban mining, these locations hold immense importance in unlocking hidden treasures embedded within our cities. The concept of urban mining embraces the idea of extracting valuable resources from urban areas, and the remnants of past metallurgical activities serve as prime targets for this sustainable practice.

However, the legacy of these metallurgical sites is not without its challenges. Years of industrial activity have left a mark on the land, as soil pollution became an unfortunate side effect. Heavy metals and other contaminants seeped into the earth, posing risks to both human health and the environment. It is imperative, therefore, to address this pollution and rehabilitate these sites, not only for the sake of the present but also for the future well-being of our communities.

Moreover, in a region where land availability is at a premium, the pressure to optimize land use is ever-present. The rehabilitation of these abandoned metallurgical sites offers a compelling solution. By repurposing these brownfield areas, we can alleviate land pressure, transform them into sustainable spaces, and breathe new life into the surrounding communities. Such endeavours align with the principles of sustainable development and the pursuit of a circular economy, where resources are conserved, and waste is minimized.

In this guidebook, the project partners of NWE-REGENERATIS demonstrate novel site characterization methods and tools to boost the initiation of resource recovery projects on former metallurgical sites and deposits in North-West Europe. By doing so, the project NWE-REGENERATIS takes a step closer towards reclaiming our industrial heritage, preserving the environment, and building a future that thrives on the principles of sustainability.

The project NWE-REGENERATIS and this guidebook were made available by the European Regional Development Fund of the European Union. All activities were funded by Interreg North-West Europe (NWE) under the thematic priority of resource and materials efficiency. Interreg NWE is a European Territorial Cooperation programme to promote a green, smart and just transition for all NWE territories with the aim to support a balanced development and make all regions more resilient.



1 OBJECTIVES OF NWE-REGENERATIS FOR INNOVATIVE RESOURCE AND MATERIALS EFFICIENCY

1.1 TURNING POTENTIAL CONTAMINATED SITES INTO OPPORTUNITIES

Metallurgical sites and deposits in North-West Europe have played a significant role in the region's industrial history. These sites were primarily involved in the extraction, processing, and refining of metals such as iron, copper, lead, zinc, and tin. In North-West Europe, particularly countries like Belgium, France, Germany, and the United Kingdom, have a rich history of metallurgical activities. These activities were concentrated in areas with access to mineral resources, waterways for transportation, and a skilled workforce. Metallurgical sites can be found across the region, with clusters in industrialized regions like Wallonia in Belgium, the Midlands in the United Kingdom, Haut-de-France in France and the Ruhr Valley in Germany. These areas were characterized by the presence of coal and iron ore deposits, which were essential for the metallurgical processes.

Metallurgical sites and deposits hold significant importance in the context of urban mining. Urban mining refers to the process of recovering valuable resources from urban areas, including abandoned industrial sites. These sites contain substantial amounts of metals that can be extracted and recycled, reducing the need for primary resource extraction and minimizing environmental impact. However, many historical metallurgical sites and deposits are associated with soil pollution due to the release of heavy metals and other contaminants during the industrial processes. These pollutants can pose risks to human health and the environment. Therefore, rehabilitating these sites is crucial to remediate soil pollution and prevent further contamination of surrounding areas.

In densely populated regions like North-West Europe, land availability is often limited. Rehabilitating and repurposing former metallurgical sites can help alleviate land pressure by converting these brownfield areas into productive and sustainable spaces. This can include activities such as urban development, green spaces, or renewable energy installations.

Overall, the rehabilitation of past metallurgical sites and deposits in North-West Europe is important for addressing soil pollution, mitigating land pressure, and unlocking valuable resources through urban mining. These efforts contribute to sustainable development, environmental protection, and the transition towards a circular economy by optimizing the use of existing resources. If the health and safety standards are met, preservation of industrial heritage should be considered.

However, such efforts are facing major technical, environmental, economic and social challenges. The considered metallic waste streams such as aggregated materials with high ferrous metal content, scrap metals, white slags and other streams are seen as a source of pollution that are expensive to manage and eliminate. Conventional soil pollution treatments currently focus on the decontamination or landfilling of dumped materials and are not oriented towards the extraction and recovery of valuable raw materials.



Moreover, the current lack of reliable and coherent data about the economic resource recovery potential of past metallurgical sites and deposits (PMSD) and about the economic and technical viability as well as the yield of metals extraction processes is a major challenge.

Thus, the project "REGENERATION of Past Metallurgical Sites and Deposits through innovative circularity for raw materials" funded by Interreg-NWE addresses those challenges and barriers to provide solutions for the remediation of contaminated PMSD in North-West Europe.

During the project lifetime, NWE-REGENERATIS developed cost-effective methods focusing on the identification, extraction and recovery of raw materials, which are required to obtain an estimate of the sites' recovery potential. An evaluation of the feasibility and economic potential for raw material recovery and site remediation using innovative characterisation and decision support tools was developed by using data from nine pilot sites. Therefore, NWE-REGENERATIS increases the reuse of materials from PMSD in NWE, turning them into opportunities for stakeholders managing or providing environmental services for PMSD.

This guidebook is provided to all stakeholders, responsible for managing, remediating or redeveloping PMSD, which are searching for further raw materials recovery from such sites, like metal and mineral extraction, re-use of soils, and eco-catalyst production.

NWE-REGENERATIS offers the REMICRRAM methodology (REGENERATIS Methodology for Innovative Circularity to Recover Raw Materials (REMICRRAM)) promoting harmonisation of brownfields inventories in NWE, with a focus on raw materials, in order to support new business models, through a screening tool (Phase 1 – SMART PHOENIX) and decision support tool (NWE-Smart Decision Support Tool (NWE-SMARTX)) based on artificial intelligence algorithms. Moreover, an innovative investigation and characterisation solution using geophysics is provided as a main output.

The guidebook aims to mediate the major findings, methods and tools of the project NWE-REGENERATIS in a comprehensive summary with further links to more detailed reports and experts' information on the projects' <u>e-library</u>. Stakeholders shall get an overview on how to initiate recovery projects on PMSD, with information on legal and technical barriers, planning activities, solutions for historical data collection and site characterisation based on data harmonisation and non-invasive geophysical site inspection. The guidebook provides the reader with the necessary tools to initiate a recovery project on PMSD, and mediates recommendations as well as valorisation options. In three case studies the project partners of NWE-REGENERATIS provide insights into aspects and results from pilot studies on PMSD.

This guidebook is intended for a broader stakeholder audience from past metallurgical sites and deposits, ranging from site owners and site managers to environmental and valorisation service providers. For those who would like to connect with other stakeholders in the field of PMSD, the <u>open access platform</u> of NWE-REGENERATIS is highly recommended. NWE-REGENERATIS's ambition is to provide a powerful network of stakeholders and to boost the launching of resource recovery projects that will achieve tangible advances in the resource recovery sector. Further knowledge can be gained in the <u>NWE-REGENERATIS e-learning tool</u>.



2 REGENERATIS METHODOLOGY FOR INNOVATIVE CIRCULARITY TO RECOVER RAW MATERIALS (REMICRRAM)

2.1 INTRODUCTION

Europe is highly reliant on other nations in the world in order to satisfy its expanding ferrous and non-ferrous metal needs. Through the (re)mining of currently extant potentially contaminated sites abandoned by the metallurgical industries, some of these metals can be recovered and produced in Europe. Urban mining of Past Metallurgical Sites and Deposits (PMSD) opens up new opportunities for sustainable waste management, land and material recovery, human health protection and environmental risk reduction. Even though the social and environmental benefits of the urban mining projects have been assessed, stakeholders are still often reluctant to start the projects due to profitability risks associated with the lack of reliable data. Moreover, historical studies are important to conduct as they can provide valuable information regarding past industrial activities that were carried out on site. However, current traditional historical studies are more oriented towards environmental and health risks and do not consider the data collection to assess economic and valorisation potential of material deposits present on site. Thus, the NWE-REGENERATIS project has develop a new method to conduct historical studies on PMSDs, with a focus on potential resource identification on PMSD but still following the existing approach oriented environmental and health risks. The performance report on historical studies summarizes the innovative approach.

To facilitate the implementation of this kind of urban mining projects, the NWE-REGENERATIS project partners have developed the REGENERATIS Methodology for Innovative Circularity to Recover Raw materials from PMSD while regenerating the polluted sites, in short called REMICRRAM to support the new circular economy for secondary raw materials recovered from brownfields and PMSDs.

This innovative methodology helps the stakeholders to take a decision whether "to start or not to start" a valorisation project on a given site or PMSD based on the provision of best valorisation options. It also facilitates the stakeholders' decision by identifying the other drivers, like economic, social and environmental circumstances.

The objective of the REMICRRAM methodology is to encourage and guide those who are interested in revalorising PMSDs (e.g. brownfield owners, project managers, local authorities), to fully characterize a PMSD site and its economic potential for the recovery of materials, metals, soil and land.

The realisation of the innovative REMICRRAM methodology is based on modern, adapted and optimised technologies to manage PMSD. Corresponding benchmarks can be downloaded from the <u>NWE-REGENERATIS e-library</u>.



2.2 REMICRRAM METHODOLOGY

The REMICRRAM methodology consists of 3 phases:

- Phase 1: A quick screening tool to evaluate the valorisation potential of a PMSD for the recovery of materials, metals, soil and land (<u>SMART PHOENIX</u>). In case of high potential, the user can proceed to the next phase;
- Phase 2: A Decision Support Tool (DST) based on artificial intelligence and algorithms to choose the best valorisation options for materials and metals present on site (<u>NWE-SMARTX</u>);
- Phase 3: The <u>structure of an evidence-based business case</u> in order to facilitate a cost benefits analysis focusing on the efficiency, effectiveness, economic, social and environmental potential of a site-specific urban mining project.

After applying the REMICRRAM methodology (Figure 1), the user decides whether or not to initiate an urban mining project on the site. At this point, the user will have different scenarios for the site valorisation. This step helps the user to choose the best valorisation option for the site or its materials by identifying other drivers such as: biodiversity, green energy, ecosystem services, social benefits, economic benefits, environmental revenues, etc.

REMICRRAM

REGENERATIS Methodology for Innovative Circularity to Recover RAw Materials from PMSD



Figure 1: REMICRRAM methodology workflow from a database to the selection of a profitable urban mining project.



2.2.1 Phase 1 – SMART PHOENIX

This easy to use tool is destined to allow the stakeholders to evaluate the valorisation potential of a site/PMSD. The tool can be applied on one or several sites, in order to prioritize them based on their potential for materials and land reclamation. The user has to answer 16 multiple-choice questions related to the characteristics of a site/PMSD. These questions were selected to be user-friendly. Each answer of these questions is associated with a weight/ score, while the total score is obtained by adding up the scores obtained from the 16 questions. In parallel to that, the user is also asked to respond on the degree of confidence for each answer that is given. The user will be notified of the potential non-accuracy of the score in case of insufficient confidence.

The total scores of these 16 questions determine the best valorisation options for a site/PMSD, which are divided into 4 main categories: metal recovery, mineral recovery, soil enhancement for eco-catalyst production and production of eco-catalyst (see Chapter 6 Valorisation Options). High scores in any of these categories indicate that the site has potential for valorisation, and it requires more investigations through phase 2. Low scores indicate that the site is of no specific interest and there is no need to investigate it further in phase 2, which will appear due to insufficient and unreliable information. The user can verify and modify the answers of the questions and see if the new scores will recommend proceeding to phase 2.

For more details on the screening tool <u>SMART PHOENIX</u> the reader is referred to the explanatory <u>SMART PHOENIX-guidebook</u>.

2.2.2 Phase 2 – NWE-SMARTX

In case of getting high score in one of the valorisation categories mentioned in phase 1 (SMART PHOENIX), the user can proceed to a decision support tool in phase 2 (NWE-SMARTX). In this phase the user can assess the site's valorisation potential in detail and identify some technical and economical thresholds associated with the site, the deposit or the process. More details on the smart decision support tool NWE-SMARTX are given in Chapter 4.

<u>NWE-SMARTX</u> is an open-source software, developed with the data from 9 pilot sites of the project located in UK, Belgium and France. It is recommended to have an expert's assistance while using this tool due to the detailed input information needed for a reliable output and recommendation on the valorisation of the site. NWE-SMARTX indicates the best valorisation options for metals and materials based on decision trees and algorithms related to civil engineering methods, mineral processing, metallurgical extraction processes and eco-catalyst production on site (see Chapter 6).



2.2.3 Phase 3 – Business Case

After applying the smart decision support tool NWE-SMARTX, the user can choose to develop a business case for a specific site based on the recommendations received. A structure and example of a business case are available in the <u>e-library of NWE-REGENERATIS</u>. This phase helps the user to assess the economic viability of such a project based on the data provided in phase 1 and 2. It provides a realistic cost-benefit analysis with a detailed consideration of all the associated risks and affecting factors. It provides a record of the return on investment from a financial perspective and summarizes all the benefits delivered directly and indirectly. This business model structure is a management tool for an evidence-based and transparent decision-making process for brownfield owners/managers, municipalities, public/private or any other interested stakeholder to analyse the economic potential of an urban mining project before launching it. Similarly, the quantification of costs can be done for several scenarios, for example, by comparing the costs of traditional rehabilitation works on polluted sites to those of remediation using the recovery options proposed by REMICRRAM.

2.3 METALLURGICAL SITES INVENTORY STRUCTURE FOR POLLUTED SITES

The lack of a standardized framework for making economically informed decisions on launching raw material recovery projects on Past Metallurgical Sites and Deposits (PMSD) presents a significant challenge. Current inventories for PMSD were rather created to contain information useful for the rehabilitation of these sites (remediation, environmental aspects, history, etc.), but they did not necessarily address the potential of these sites for the recovery of secondary materials (<u>Benchmark report of existing inventories structures in NWE regions</u>). Moreover, traditional methods used to assess viability are expensive and require costly analyses and sampling, which further complicates the process. All these aspects make it difficult for stakeholders to assess the suitability of their site for material recovery projects.

To address this challenge, it is necessary to establish a suitable inventory that collects all the key parameters relevant for recovery projects. The MEtallurgical Sites Inventory Structure (<u>NWE-MESIS</u>) is an inventory structure developed by the NWE-REGENERATIS project. It is intended to be used directly as a structure to create an inventory or to supplement an existing one. It contains crucial parameters for developing material recovery projects from PMSD and includes some parameters that are also part of the REMICRRAM tools.

NWE-MESIS is a valuable resource for stakeholders who are considering launching recovery projects on PMSD, as it includes historical studies, site visits, pre-investigation estimates, and other relevant data. However, NWE-MESIS does not contain any datasets or analysis results. The decision to launch a recovery project depends on various drivers, such as economic, environmental, and social factors, all of which are included in <u>NWE-MESIS</u>. More information can be found in the <u>NWE-MESIS guidebook</u>.



3 SITE INVESTIGATION - APPLICATION OF NON-INTRUSIVE METHODS TO CHARACTERISE PAST METALLURGICAL SITES

3.1 CONTRIBUTION OF GEOPHYSICS TO THE DEVELOPMENT OF A RAW MATERIALS AND POLLUTION DISTRIBUTION MODEL (RAPIDM)

This chapter presents a proposed workflow for the use of geophysical survey techniques to characterise PMSD in order to guide their future regeneration. Geophysical survey techniques provide many advantages over traditional "intrusive" studies and should always be considered at the outset of a project when trying to establish the resource potential of an existing PMSD.

Geophysical survey methods may be used to enhance the characterisation of PMSD sites, in terms of the spatial extent/volume and/or the composition/distribution of metallurgical materials across a site. Rather than providing a detailed description of individual techniques (e.g. see <u>benchmark on geophysical investigation report D 1.3.1</u> and <u>performance report on new geophysical characterization method</u>), this chapter presents a high-level approach for the design of a geophysical survey through the development and subsequent improvement of a conceptual ground model. The final aim of the process described is to construct a Raw Material and Pollution Distribution Model (RAPIDM) which describes the spatial/volumetric distribution of indicative parameters of the metallurgical materials, at a scale suitable to assess the economic viability of potential PMSD mining operations.

Geophysical prospecting methods are rapid, non-invasive, surface-based techniques, used to measure bulk ground properties, such as electrical conductivity (or its inverse, electrical resistivity), density or stiffness. The most effective use of geophysical surveying relates to the ability to investigate relatively large areas, in order to delineate (map) areas of contrasting material properties. In addition, geophysical methods are largely non-invasive and do not present the same risk of cross-contamination or damage to contamination barriers associated with conventional invasive sampling such as trial pitting and drilling.

Geophysical surveying can capture much greater information concerning spatial heterogeneity across a site, or vertically, and is more cost effective than point measurements alone (e.g. intrusive boreholes/trenches or point sensors). For example, to identify anomalies of a minimum area of 25 m² (at ~1 m depth) with confidence within a site of dimensions 100 * 100 m, using intrusive methods alone would require over 600 trial pits of 1 m² to be dug - a significant cost, both financially and in terms of the time/resource required. In comparison, multiple geophysical mapping techniques could be undertaken across the site in a fraction of the time and at vastly reduced costs. Specific areas selected using geophysical imagery could then be chosen for verification through a small number of targeted sampling (e.g. boreholes, trenches). The combination of geophysical imagery and ground truth allows to identify the PMSD's extent and structure, as well as to quantify changes in metallic content of the different deposits. The NWE-REGENERATIS proposed workflow to set-up a geophysical survey from the data collection to the interpretation and validation is summarised in Figure 2.



3.2 PRINCIPLES ON HOW TO USE GEOPHYSICS

A correct application of a geophysical survey is critical in order to achieve reliable results. First, it should be kept in mind that geophysical methods are indirect techniques and the measured physical property might point to different possible interpretations. As an example, high conductivity indicates both, increased clay or metal content. Therefore, in order to reduce this uncertainty, it is highly recommended to apply a combination of complementary geophysical methods, which measure different and unrelated bulk ground properties. Furthermore, especially due to the highly heterogeneous structure of PMSD, it is mandatory to use targeted intrusive samples both in order to verify the geophysical results and to calibrate the geophysical processing and modelling.



Figure 2: Main steps of the suggested workflow on how to use geophysical techniques to build a RAPIDM.



A geophysical technique can only detect a target if it is large enough in comparison to the geophysical method resolution, and causes a significant contrast in the measured material property. Conductivity maps measured with electromagnetics for example, will only efficiently delineate a metallic body if the conductivity contrast between the slag and the host material is high enough. However, the sensibility of geophysical methods decreases with depth, meaning a geophysical anomaly might be detected at the surface, while it remains invisible at depth.

Furthermore, every geophysical method has different advantages and limitations. Some methods are better to map lateral changes whereas other methods are able to measure up to greater depths. The required time and staff for geophysical acquisition and processing can vary significantly. Table 1 gives an overview of possible applications of main near-surface geophysical methods for PMSD characterisation together with the staff required to deploy them, acquisition and processing times. However, the correct choice of methods is site dependent. Therefore, it is crucial to choose the primary geophysical technique(s) and the associated measurement parameters based on a priori knowledge of the site conditions (expected heterogeneities in material properties) and the objective/target of the survey.

Table 1: Suitability of geophysical methods for different applications related to PMSDs study. Abbreviation list: EMI – Electromagnetic induction, MAG – Magnetometry, ERT – Electrical resistivity tomography, IP – Induced Polarization, MASW – Multi-channel analysis of surface waves, SRT – Seismic refraction tomography, GPR – Ground penetrating radar, HVSRN – Horizontal to vertical spectral ratio of noise, SP – Spontaneous potential, GRA – Gravimetry.

		Мар	oing				Profil	ing			
		EMI	MAG	ERT	IP	MASW	SRT	GPR	HVSRN	SP	GRA
	Lateral extent										
DMSD structure	Cover Layer thickness										
PMSD Structure	Vertical extent										
	Buried utilities										
PMSD	Slag zonation										
characterisation	Metallic content										
Environmental	Host material										
conditions	Groundwater table										
Staff req	uired for survey	ţţ	Ť	ţţ	ţţ	Ť.Ť.Ť	Ť.Ť.Ť	\neq	Ť	††	Ť
Required time for survey		(1)	Ð	ĊĊ	ĊĊ	CDD	DDD	()	(1)	ĊĊ	CCC
Required ti	Ŀ	⊕	ĊĊ	ĊĊ	CDD	CDD	CCC	Ŀ	ĊĊ	ĊĊ	

Primary method

May be used but not the best method

Unsuited

Guidebook for the REGENERATIS methodology Site Investigation - Application of Non-Intrusive Methods to Characterise Past Metallurgical Sites



3.3 GEOPHYSICAL SURVEY

A priori information and identification of knowledge gaps

A first step in the planning of a geophysical survey is the collection of already available a priori site information. This information can range from PMSD extent, structure (e.g. presence of metallic scraps or HDPE membrane) to deposit composition and can come from various sources. The available information should allow for creating a preliminary conceptual model of the PMSD as shown in Figure 3. Depending on the available information, this conceptual model can vary in the degree of detail. Based on the conceptual model, "knowledge gaps" can be identified and the objective/target of the geophysical survey can be defined. Furthermore, the conceptual model is vital in order to choose the appropriate combination of geophysical techniques, delineate areas of interest, and define required survey resolution and parameters.



Figure 3: Example of a conceptual model built from a priori site information, including information such as PMSD extent, host material, waste body, cover layer, groundwater table or potential contamination. Site constraints for future geophysical survey should also be identified.



Geophysical characterisation of past metallurgical sites and deposits

The effectiveness of any geophysical survey will relate to the overall size of a field site and scale of heterogeneity encountered across that site. A survey should be designed to capture information at a scale appropriate to characterise any variations in material properties throughout a field site, or to resolve anomalies/features of a desired size/volume. Survey planning should therefore take into account any pre-existing information regarding the site, such as construction information, records of deposit and any intrusive information that may have been collected previously, as well as specific site constraints such as accessibility or presence of disturbing structures (e.g. metallic objects as illustrated in Figure 3). This is particularly important for deciding which techniques may be of most significance (see list of geophysical technics in Table 1).

The choice of primary geophysical technique(s) used will likely relate to the type of property of interest, i.e. if a particular material such as metal or plastic is to be recovered preferentially, but it is recommended that multiple techniques should be applied, preferably measuring a range of different and unrelated properties. Changes in the bulk properties of PMSD materials relating to variation in the types of slag deposited (e.g. low resistivity = high clay content or high metal content) may mean that a particular technique (e.g. electrical or electromagnetic) is most appropriate, but a secondary technique (e.g. relating to material stiffness or density), may help to further refine the interpretation of the collected geophysical dataset.

A direct result of using a number of geophysical methods, which measure different but complementary physical properties, calibrated with targeted sampling, is the significant increase in the level of confidence placed in any final interpreted RAPIDM, associated with the reduction of investigation costs. Geophysical methods, when correctly applied, help to better understand PMSD. Combined with a priori information and targeted sampling, they can help to identify the PMSD extent and to characterise and identify changes in deposit composition.



Figure 4: Examples of mapping (left) and profiling (right) geophysical methods. The geophysical mapping method illustrated is the electromagnetic induction (EMI) which uses electromagnetic fields to measure electrical conductivity and magnetic susceptibility of the subsoil. The geophysical profiling method shown is the electrical resistivity tomography (ERT) which allows to image (in 2D or 3D) the electrical resistivity distribution of the subsoil by injecting electrical current and measuring the resulting difference in electrical potential via buried electrodes.



The following paragraphs introduce a suggested process through which a geophysical survey may be designed. The types of existing data discussed are not an exhaustive list and any source of information available should be utilised where possible. Geophysical methods can be divided into two categories (see Figure 4):

- **Mapping methods:** These methods provide a rapid overall knowledge of extent, structure or lateral changes in composition or thickness of cover and metallurgical deposit layers.
- **Profiling methods**: These methods are less cost effective but provide more detailed information about changes with depth. High-density surveys can be combined to create a 3D model of the PMSD.

Often mapping methods are applied first in order to gain an overall view of the PMSD extent and spatial structure. The profiling methods are generally used afterwards for more detailed studies. Either they are used for targeted investigations on areas which require additional information based on a priori information or/and on the results of the mapping methods. Or they can be used as single profiles gaining an overview of the depth/structure of the PMSD. A proposed workflow is schematically illustrated in Figure 5 and Figure 6 on a synthetic case study.



Figure 5: Application of mapping methods to the synthetic case study. The spatial coverage of electromagnetic induction (EMI) and magnetometry (MAG) is shown on the left. The survey is generally carried out along a grid with an interline spacing that should ideally not be too coarse to capture site heterogeneity. After data processing, the results can be displayed as maps showing the physical properties targeted by the methods (here electrical conductivity for EMI and magnetic field anomaly for MAG). In this case, it is possible to estimate the lateral extent of the PMSD and to identify two areas related to the different types of slag material.



Mapping methods (EMI and MAG) are at first used to delineate the lateral extent of the PMSD and gain an overview of lateral heterogeneities within the PMSD. Once the lateral zonation is evidenced, the vertical extent of the PMSD and the deposit zonation can be investigated by applying profiling methods (ERT and IP). The two profiles image the deposits by a conductive (**resistivity**) and chargeable (**chargeability**) model. In the upper part of the PMSD, the model resolution at depth is not sufficient to reliably estimate the total thickness of the PMSD, but the homogeneity of the observed electrical properties (resistivity and chargeability) suggests the presence of a single type of slag. In the lower part, the model resolution and electrical contrast are sufficient to map the interface between the host geology and the slag at depth. Given the observed electrical signatures of two different types of metallurgical deposits. The shallower is conductive and chargeable, while the second one is not chargeable. This latter could correspond to inert metallurgical deposits characterized by low metallic content.

Calibration and validation through targeted sampling

The joint interpretation of geophysical data, together with the prior knowledge of the investigated PMSD, allows for updating the conceptual site model (Figure 3). Still, some uncertainty may remain, given the indirect nature of information provided by geophysics. To validate and calibrate the conceptual site model, ground truth data are generally required. A site dependent sampling plan based on the conceptual site model should target the following:

- 1) Areas characterised by different geophysical signature ;
- 2) The background conditions;
- 3) Areas where geophysical methods cannot provide any or enough coverage.



Figure 6: Application of profiling methods (electrical resistivity tomography – ERT and induced polarization – IP) to the synthetic case study. Such methods are generally applied along 2D lines (see left figure) or 3D grids. They provide information about the vertical and horizontal zonation of the PMSD. When only applied at the soil surface, the resolution of such methods generally decreases with depth making the interpretation of deep structures difficult (see resistivity and chargeability models corresponding to the upper profile).



At least one sample should be collected in each of the aforementioned zones. Increasing the number of samples collected in each of these zones will improve the statistical robustness of the resulting ground truth data. If the sampling plan allows for a large number of samples to be collected, statistical tools such as Latin Hypercube Sampling informed by the geophysical data could be considered (more information in <u>reference [3]</u>).

After the joint interpretation of geophysical and sampling data, it must be decided whether the information available on the site is sufficient to establish a reliable RAPIDM or whether further studies are necessary in which case it is possible to iterate on geophysical and sampling investigations as illustrated in the proposed workflow (Figure 2).

3.4 BUILDING OF A RAW MATERIALS AND POLLUTION DISTRIBUTION MODEL (RAPIDM)

Building a RAPIDM is the final step of the proposed workflow (Figure 2). It is a very important one because it should allow for assessing the economic viability of potential PMSD regeneration operations via the developed <u>NWE-SMARTX</u> tool (see dedicated chapter 4 Smart Decision Support Tool NWE-SMARTX for more information). The delivered RAPIDM should contain the spatial/volumetric distribution of indicative parameters of the metallurgical materials. In NWE-REGENERATIS, we followed mainly two approaches to build the RAPIDM.

The first one is relatively simple and consists of building the RAPIDM by "visually" comparing geophysical and ground truth data. In such an approach, ground truth data are used to constrain the model whereas geophysical data are used to ensure spatial continuity. Such an approach is illustrated in the synthetic case presented in Figure 7.

The second approach uses co-located geophysical and sampling data to produce models of the probability of belonging to a predefined slag facies. Such an approach offers the advantage to provide models that take into account uncertainty and loss of resolution occurring with depth, but are somewhat more complex to produce than those provided by the first approach (see chapter 7.1 Duferco case study). For more information on the probabilistic approach, the reader is referred to reference [4] and to the report on RAPIDM for the 3 pilot sites of the NWE-REGENERATIS project. For both approaches, multivariate clustering methods could be applied prior to building the RAPIDM with the aim of reducing the number of dimensions of the geophysical dataset to be compared with the ground truth data.







Figure 7: RAPIDM of the site obtained using the first approach. The proposed model provides a spatial view of the different slag facies identified together with their estimated volumes. The mass of the metallurgical deposits is estimated based on the slag density (measured in laboratory or in the literature) and the volume of metallurgical deposits given by the RAPIDM. Other relevant information such as the presence of a groundwater table, the composition of the slag facies or the presence of contamination may also appear in the RAPIDM.



3.5 TAKE HOME MESSAGE

Geophysical survey techniques provide many advantages over traditional "intrusive" studies and should always be considered at the outset of a project when trying to establish the resource potential of an existing PMSD.

Advantages of Geophysics:

- **Fill gaps** between existing boreholes or other ground truth.
- Personal less exposed to potential hazardous material as a non-intrusive method
- Cost effective & rapid

Having planned and executed a geophysical survey based on the conceptual site model developed from the existing data, it should be possible to refine the model with regard to the overall extents and any structure within the PMSD, as well as to attach measured (or modelled) geophysical parameters to estimate the distribution of recoverable materials throughout the asset, transforming the conceptual site model into a RAPIDM, including data acquired from subsequent intrusive surveys where appropriate.

Geophysics provides specific information to assist in providing a RAPIDM:

- PMSD extent (depth and lateral extent)
- PMSD structure (clay cap thickness, HDPE membrane, clay boundaries etc.)
- Changes in slag deposit composition
- Position of buried utilities

As a final note **regarding the validity of a RAPIDM derived from geophysical data**, **any interpretation should always have some element of ground-truthing through intrusive sampling**. Also, it should be borne in mind that the geometry of any survey will necessarily limit the scale of the RAPIDM and at which the spatial heterogeneity is captured. For this reason, when constructing a RAPIDM, it is important to not over-interpret areas covered by sparse data, where the potential exists to miss rapid lateral changes between data points/profiles. As such the level of confidence in the RAPIDM must be estimated to include any uncertainty associated with survey geometry.

3.6 REFERENCES

- 1. Reynolds, J.M., An introduction to applied and environmental geophysics. 2011: John Wiley & Sons.
- 2. Soupios, P. and D. Ntarlagiannis, Characterization and Monitoring of Solid Waste Disposal Sites Using Geophysical Methods: Current Applications and Novel Trends, in Modelling Trends in Solid and Hazardous Waste Management. 2017, Springer. p. 75-103.
- 3. Minasny, B. and A.B. McBratney, A conditioned Latin hypercube method for sampling in the presence of ancillary information. Computers & geosciences, 2006. 32(9): p. 1378-1388.
- 4. Hermans, T. and J. Irving, Facies discrimination with electrical resistivity tomography using a probabilistic methodology: effect of sensitivity and regularisation. Near Surface Geophysics, 2017. 15(1): p. 13-25.



4 SMART DECISION SUPPORT TOOL (NWE-SMARTX)

In case of getting high score in one of the valorisation categories mentioned in the first phase of site potential investigation (Chapter 2.2.1) and good score for the confidence level, the user can proceed a second phase with more technical data as inputs with <u>NWE-SMARTX</u> decision support tool. In this phase the user can assess the site's valorisation potential with more details and identify some technical and economical thresholds associated with the site.

NWE-SMARTX is an open-source software, developed with the data from pilot sites of the project located in UK, Belgium and France and additional data from other sites and expert knowledge. NWE-SMARTX indicates the best valorisation processes and treatments for metals and materials based on decision trees and algorithms related to civil engineering methods, mineral processing, metallurgical extraction processes and bio-based catalyst production.

4.1 DESIGN OF THE SMART DECISION SUPPORT TOOL NWE-SMARTX

4.1.1 Why and How Artificial Intelligence can be used for PMSD remediation.

Defining a PSMD remediation strategy is a complex challenge and requires a large panel of expertise to be applied to solve not only issues with logic rules but also issues where no equation can be applied. A simple question such as 'is the soil fertile for ray grass' is not so simple when the soil includes metal contamination. What are the parameters to be used to define fertility? What is toxic for the targeted vegetal? What are the limit values?

The definition of possible technical options to be deployed or applied for metal or mineral recovery is also a question with answers depending on multiple conditions with no clear threshold between the good and the wrong answers.

To face such challenges, Artificial Intelligence (AI) provides supports by imitating experts experience and learning from experience.

Learning from experience for algorithm is the AI training: using various experiences providing 'description data' of initial material or situation (=input parameters) and the 'result information' (=output parameters) describing the material after processing this initial material or describing which technical solutions are adapted, we can generate datasets to develop and improve models able to estimate a 'result' from a full or partial 'description'.

NWE-REGENERATIS project provided site descriptions, soil descriptions, sample descriptions and associated results for pertinence to use various techniques or performance results evaluation. This has been done in 5 specific domains of expertise to be considered for PMSD remediation strategies to provide decision-making support information. Partners created datasets with 'inputs' conditions and associated 'outputs' results or valid option for each domain of expertise. Datasets have been stored in a project database to develop and train algorithms for an AI based decision support tool: <u>NWE-SMARTX</u>.



4.1.2 Ambition of NWE-SMARTX

NWE-SMARTX is made to be used once general information collected confirms site's potential for material/metal recovery from this PMSD (<u>SMART PHOENIX</u> see chapter 2.2.1). NWE-SMARTX assess the site's valorisation potential on the basis on some selected key technical information related to various technical domains to provide site managers with key orientation to be prioritized.

NWE-SMARTX aims to provide site managers with a first level of technical information for recovery and circular material economy NWE-SMARTX provides projects. orientations after an AI based first level of on complete input expertise or partial information in the following domains:



technical Figure 8: Database main organisation with Inputs & level of Outputs data for each domain

- a. Geophysical technics to be applied to map and evaluate recovery material potential
- b. Excavation technics to be used
- c. Mineral processing for mineral/metals recovery
- d. Metallurgic processes for metals extraction from waste
- e. Soil improvement potential to recover soil fertility and to generate innovative bio-based catalyst from biomass growing on contaminated soils (=eco-catalyst)

With NWE-SMARTX results, the site manager will have confirmed options for deeper investigation together with experts for a specific project design.



Figure 9: NWE-SMARTX ambition: improving site potential evaluation with AI algorithms.



4.1.3 NWE-SMARTX design process

Key input parameters have been determined for the domains of excavation techniques, geophysical techniques, metal recovery, mineral recovery and soil fertility for eco-catalyst production. Moreover, expected indicators for those 5 domains were used as outputs.

Datasets were created for each of the 5 domains with points of views from experts and scientific publications data to include knowledge available in the scientific and experts' community. This has been completed thanks to project sites investigations with specific lab scale tests as well as onsite tests done during the project.

Scientific publications, expert's opinion, site specific data, soils/material samples, as well as onsite demonstrations provided initial condition description data (inputs) and conclusions (outputs) to define, train, and test AI based algorithms able to replicate experts' opinions or evaluate future results from new input parameters.

Various algorithms were developed and tested for each 'output' indicator:

- K-Nearest Neighbours
- Random Forest
- Naive Bayes
- Decision Trees
- Support Vector Machines
- Logistic Regression



Figure 10: Various algorithms developed and tested for each output indicator.



In a first step, algorithms have been defined, trained and tested on data from experts' opinion, scientific bibliography, and first projects lab scale tests results. In a second step, algorithms have been improved, trained, and tested with data from project lab scale tests and on-site demonstrations.



Figure 11: Example of data used for main steps of algorithms definition, improvement and training

For each output indicator, the most accurate algorithm has been selected regarding its accuracy evaluation. The selected algorithms and related database have been packed in an easy-to-use application: <u>NWE-SMARTX</u>.



	Crushing	Screening	Mag. Separation	Grav. Separation
K-Nearest Neighbor	96%	65%	65%	88%
Naive Bayes	69%	69%	77%	92%
Random Forest	88%	77%	58%	96%
Decision trees	96%	81%	69%	96%
SVM	85%	85%	69%	78%
Logistic Regression	92%	73%	58%	100%
	L		γ	
	KNN	SVM	Naive Bayes	Logistic Reg.

Figure 12: Example of NWE-SMARTX results for mineral processing output indicators and selected algorithms

4.2 NWE-SMARTX PERFORMANCE

NWE-SMARTX performance has been evaluated based on an available dataset when this guidebook was written. The following performance evaluation is mainly done with DUFERCO project site (see Chapter 7.1) input data extracted from REGENERATIS dataset to benefit a full set of data. This section compares NWE-SMARTX forecasts with experts' opinion or lab test values with raw results and performance evaluation. Those results are site specific and are to be considered as indicators of the capacity of NWE-SMARTX for a future wider use.

Geophysical investigations: adequate techniques to be used (%)

	GPR	ERT	IP	SRT	MASW	EMI	Mag
Expert opinion	20%	100%	100%	100%	100%	60%	60%
NWE-SMARTX forecast	20%	100%	100%	100%	100%	60%	70%

→ 100% of correct predictions with minor deviation in one technique

Excavation techniques: adequate technique to be used (Yes =1 / No=0)

	prevention plan for worker safety	hydraulic breaker not required	hydraulic breaker required	drainage and water treatment	direct digging	ripping before digging	hammer before digging	blasting before digging	stability check
Expert opinion	0	1	0	0	1	0	0	0	1
NWE-SMARTX									
forecast	0	1	0	1	1	0	0	0	1

→ 100% of correct forecast with minor deviation in one technique



Mineral processing: adequate technique to be used (Yes =1 / No=0)

		Crushing	Screening	Magnetic separation	Gravimetric separation	Electrostatic separation	Pelletizing	Washing	Cement	Sub base	Thermal plasma	Wet curing	Recycling material	Soil improvement	Geopolymer synthesis	CO ₂ capture
Test 1	Expert opinion	0	0	1	0	0	0	0	0	0	0	1	0	0	0	1
	NWE-SMARTX forecast	0	1	0	0	0	0	0	0	0	0	1	0	0	0	1
	Expert opinion	0	0	1	0	0	0	0	0	0	0	1	0	0	0	1
Test 2	NWE-SMARTX forecast	0	1	0	0	0	0	0	0	0	0	1	0	0	0	1

➔ 87% of correct forecast

Metallurgic processes: adequate technique to be used (Yes =1 / No=0)

		Crushing	Screening	Magnetic separation	Gravimetric separation	Densimetric separation	Eddy current separation	Electrostatic separation	Flotation	Dewatering	Agglomeration pelletizing	Sintering blast furnace	Waelz Rotary	Electric arc furnace	Other thermal process	Hydrometallurgy	Electrowinning
Test 1	Expert opinion	0	0	1	1	1	1	1	0	0	0	1	0	0	1	1	0
	NWE-SMARTX forecast	0	1	1	1	1	1	1	0	1	0	1	0	0	1	1	0
Test 2	Expert opinion	0	0	1	1	1	1	1	0	0	0	1	0	0	1	1	0
	NWE-SMARTX forecast	0	1	1	1	1	1	1	0	1	0	1	0	0	1	1	0
Test 3	Expert opinion	0	0	1	1	1	1	1	0	0	0	1	0	0	1	1	0
	NWE-SMARTX forecast	0	1	1	1	1	1	1	0	1	0	1	0	0	1	1	0
Test 4	Expert opinion	0	0	1	1	1	1	1	1	1	1	0	0	0	1	1	1
	NWE-SMARTX forecast	0	1	1	1	1	1	1	1	0	0	0	0	0	1	1	0
	Expert opinion	0	0	1	1	1	0	1	1	1	1	0	0	0	1	1	1
Test 5	NWE-SMARTX forecast	0	1	1	1	1	0	1	1	0	0	0	0	0	1	1	0

➔ 83% of correct forecast

→ 12/16 techniques with 100% of correct forecast

Soil fertility index and Zn concentration in plant for Eco-catalyst synthesis potential evaluation

	Fe	ertility index	Zn con	centration in plant
	Lab test	NWE-SMARTX forecast	Lab test	NWE-SMARTX forecast
Test 1	1,86	1,97	43,8	49
Test 2	1,87	2,33	51,7	47
Test 3	2,02	2,38	61,4	56

→ <u>Soil fertility index:</u> forecast is around 106% to 118% compared to lab tests.

→ <u>Zn concentration in Ray grass</u>: forecast is between 91% and 112% compared to lab tests.



Performance evaluations synthesis

Geophysics: investigations technics to be used	very good
Excavation techniques: adequate technique to be used	very good
Mineral processing: adequate technique to be used	good
Metallurgic processes: adequate technique to be used	good
Soil fertility: index estimation	very good
Zn concentration in plant: Eco-catalyst synthesis potential evaluation	very good

Notice: NWE-SMARTX has been developed with specific datasets related to a limited site number and AI based algorithms are trained with those datasets. Consequently, NWE-SMARTX performance is not guaranteed for any site and any condition.

4.3 How to use NWE-SMARTX

NWE-SMARTX is an easy-to-use tool where the users must only fill in the input parameters related to each domain. NWE-SMARTX interface will propose the full set of inputs to be filled in for each investigated domain, but the user can only provide partial indications to get results.

Site	Input Data Results		
DF			
Ge	ophysics Excavation Mineral Metal Ecocatalyst		
In ea	ocatalysis, metallic elements in accumulator/tolerant plants are recovered and transformed in potential for ecocatalyst on your site.	to eco-friendly plant-based catalysts. Ecocatalysis process has two advantages: soil remediation	by using accumulating pl
^	Laboratory data		
	InputName	Value	Unit
	Inventory		
	Available bareland area	5	ha
	Area easily accessible with machines	×	-
	Laboratory analyses		
	Soil contamination : Zn content (required)	3738,6	mg/kg
	Sand fraction (granulometry)	10	%
	Silt fraction (granulometry)	10	%
	Clay fraction (granulometry)	80	%
	Soil pH	8,09	-
	Cation exchange capacity	0	cmol+/kg
	CaCO ₃	123.6	g/kg
	Available phosphorus	0,81	g/kg
	Corg / Ntot	0	-
	Electrical conductivity	0	Ms/cm

Figure 13: NWE-SMARTX input data interface for soil fertility estimation and Zn-based eco-catalyst production.



Site	nput Data	Results							
OFC									
Geophys	sics Excava	ation Min	eral Metal	Ecocatalyst					
In ecocataly: the potentia	sis, metallic eleme I for ecocatalyst o	nts in accumulate n your site.	or/tolerant plants are	e recovered and tran	sformed into eco	-friendly plant-base	d catalysts. Ec	cocatalysis process has two adva	antages: soil
- Labor	ratory data								
^ Resul	ts								
Nam	ne							Value	
Soil Tex	kture							Optimal	
Fertility	(Medium	
Zn in p	lant							161,01mg/kg	
Recor	nmendation								
Prereque Soil fer	uisite: potential a rtility is very low. I	rea for ecocataly Ecocatalyst prod	st must me bare: Ne uction might not be	building attached feasible or require	or sealed surface important amen	s (covered with wa dment of the soil. T	terproof coati he cost should	ng such as roads or parking) d be integrated in the business	model.

Figure 14: NWE-SMARTX results interface for soil fertility for ray grass cultivation and estimation on Zn concentration in ray grass grown on the tested soil.

Output Results give also indications and recommendations about which geophysical prospection method is applicable on site, which civil engineering technics are adapted, which extraction process should be applied based on the materials and residues identified on site and which treatment is most suitable. Several scenarios could be applied for the same site. It's up to the user to choose one of them and then to prepare the cost benefits analyses (phase 3 of REGENERATIS methodology, see Chapter 2).



5 RECOMMENDATIONS FOR RESOURCE RECOVERY FROM PMSD

5.1 BARRIERS

5.1.1 Legal complexity

According to the JRC "To date, soil is not subject to a comprehensive and coherent set of rules in the European Union. The protection and sustainable use of soil is scattered in different community policies contributing in various degrees to mainly indirect protection of soil, for example through environmental policies on waste, water, chemicals, industrial pollution prevention, nature protection and biodiversity, nitrates and pesticides, sewage sludge, forestry strategy, climate change adaptation and mitigation, and biofuels. For soil contamination 13 different pieces of EU legislation apply (...)." (European achievements in soil remediation and brownfield redevelopment, JRC, 2017)

The approach to contaminated sites and brownfields varies across the EU and there is no specific directive for soil remediation.

The EU uses different types of legal acts to achieve its objectives. Some of them are binding some apply to all EU countries and some to just a few.

Depending on the type of legal acts, their content may or may not be directly applicable in the Member States. Only the regulation has direct effect. The directives set objectives, but leave the concrete details to the Member States, so that national legislation differs from one another. The use by the EU of these different types of legal acts further increases the legal complexity. Moreover, the objectives of the Directives are sometimes more or less lost when transposing Directives into national legislation.

5.1.2 Lack of data

An important barrier for redevelopment of PMSD is a lack of knowledge on what is actually present in the soil. Due to the historical activities, a lot of sites suffer from what is called a historical contamination. Because of a lack of data, PMSD and brownfields are often left vacant as isolated areas. In many cases there is a need to have access to all kind of data:

- Which substances are actually in the soil
- Which kind of activities were present on the site
- What could be potentially interesting materials or resources from a site
- What are the risks (health, migration, financial, ...) related to contaminations on-site
- ...



5.1.3 Short-term vision

Including PMSD or landfill sites in general it is a challenge to integrate these sites within broader ambitions. For example not only reintroducing them in the circular economy but trying to couple them towards integral spatial planning and climate policies are not evident. On a local scale actors like site owners or municipalities often lack the capacity and knowledge. Triggers for acting on a site are mostly short term, activated by a short term need that needs to be solved, for example a health hazard, a development opportunity. Rarely these sites are incorporated in a long term strategy. An integrated approach with new developments is often seen as too difficult, as a burden within the planning process that is not likely to tackle right now. So, these sites are really 'blind-spots' within the broader context.

5.1.4 Financial infeasibility

A main barrier in the implementation process is still the financial feasibility. Where on the site scale either binding legislation or a revenue path is clear, the redevelopment seems evident. Where these are lacking or more unclear, the development paths encounter obstacles. The current state of the art and the current financial valuation of raw materials make the profitability and feasibility of these concepts still insufficient to roll out as systemic solutions.

The above mentioned barriers present challenges that can be overcome by ambitions, which provide incentives for recovery projects and thus establish objectives and solutions with benefits in circular resource management, healthy environments and integrated spatial development.



Figure 15: Overcoming barriers by ambition. Challenges are faced by incentives that establish solutions.



5.2 CRITICAL SUCCESS FACTORS IN PMSD REDEVELOPMENT

To ensure long-term success, the rehabilitation of PMSD requires support from organizational and legal mechanisms. NWE-REGENERATIS conducted webinars, meetings, and excursions to discuss their findings on four main themes:

- 1. Identifying legal barriers
- 2. Offering solutions/recommendations to overcome those barriers and implement resource recovery projects
- 3. Promoting Public-Private-People Partnerships (PPPP)
- 4. Providing implementation guidance

The characterization of the PMSD plays a crucial role in the development of a resource recovery project. Insufficient data on the content and context can result in incomplete or incorrect decisions. NWE-REGENERATIS has developed a methodology called REMICRRAM (see Chapter 2.2), which aims to achieve the following objectives:

- 1. Characterize and select post-metallurgical sites and deposits with potential for raw material recovery.
- 2. Demonstrate the interest in recovering specific industrial waste deposits from polluted former metallurgical sites.
- 3. Create a new business model that enables transparent cost-benefit analysis, maximizing material and land recovery while reducing rehabilitation costs.

Three interconnected themes have been identified to structure the redevelopment process:

- 1. **Ambitions**: These provide incentives for the project and establish goals or objectives based on those ambitions.
- 2. **Planning and design process**: This encompasses all relevant steps for evidence-based project realization, including analysis, legal drafting, and construction.
- 3. **Decision-making and participation process**: Involvement of stakeholders, actors, and relevant expertise throughout the entire process is crucial for a successful resource recovery and rehabilitation project.

The interconnectedness of these three domains is essential as it establishes a link between the content (substances, materials, etc.) and context (urbanization trends, climate change, etc.) of any PMSD project.

The general critical success factors in PMSD redevelopment are closely linked with brownfield redevelopment in general and the following items should be considered according to the leadership in brownfield renewal network.

- Understand the landscape and the context where the site is located: Conduct an inventory, determine regulatory obligations and policy options, and identify key stakeholders.
- Build partnerships: Enhance relationships with pertinent local organizations, financial institutions, developers, regulators and other organizations, and create a community brownfield advisory group.


- Devise a strategy: Develop a strategy that considers what the future ambitions of the site, its content and surroundings might be. Build also programs to encourage redevelopment.
- Promote programs and opportunities: Build awareness by creating a communication plan and promotional materials and by participating in or hosting local events.
- Manage programs and projects: Administer your municipal brownfield programs, and facilitate the redevelopment of local properties.
- Evaluate, improve and celebrate: Assess and ensure ongoing improvement of policies and programs, and celebrate your successes.

5.3 RECOMMENDATIONS

Specific regulations on soil remediation offer options to deal with polluted soil even generated decades ago. In order to avoid a long time gap between the closure of activities and the start of the redevelopment process, one should consider the obligation of a soil survey at the moment of closure.

Promotion of databases entailing a broad range of information on PMSD sites (and contaminated sites in general) can prompt both governments and potential developers to take action. The NWE-REGENERATIS project offers the NWE-MESIS (see Chapter 2.2), which summarizes all data that is necessary to estimate the resource potential of a PMSD site.

Given that the redevelopment of PMSD sites are complex processes where a multi-disciplinary approach is necessary, we recommend that parties (or a party) have an overview of the 'journey' of the site on as a whole. A party that can take the role of the guide, facilitator or captain can facilitate the process. A recent good practice example is the brownfield covenant legislation in Flanders, which provides this opportunity. NWE-REGENERATIS developed a systemic framework (more details in: <u>Barriers and obstacles at legal/organizational level in the valorization of past metallurgical sites and deposits – a systemic approach</u>) that can act as a 'projective' that allows for a dialogue with the relevant stakeholders to happen in order for further integration between the different domains.

Different actors and stakeholders hold positions that are risk averse and less solution oriented. With an increased capacity for policy makers to act on a strong stakeholder management (a role as a site transition manager) more dynamic on our PMSD sites would be possible.

The current static, linear and sanctioning legislation often hinders the reintegration of PMSD sites. We hope for the openness to talk and work towards a more dynamic, circular and facilitating framework. We also recommend considering specific deviations/exemptions in the legislation to support these complex redevelopment projects. Not to fall short on the risks or potential hazards these sites often form. But an overarching law that creates a very open framework for certain contexts in response to the amalgam of legislations (soil, waste, materials, spatial, ...) could form the base for a more dynamic usage of our PMSD sites. The brownfield covenants form such an example. A unique account manager might also improve the process flow.



The concept of save storage of contaminated soil and waste, in anticipation of future technologies for recycling and reuse should be taken into consideration. We should be careful not to hypothecate recovery chances. An actual discussion should be 'where to store temporarily interesting streams of resources'. And what kind of temporal activity can we allow on certain sites. We should move away from the current idea of orphan sites, abandoned in our landscape to integrated treasuries for the future.

Contaminated soils, waste and other production residues are "materials", just like raw materials. Its reuse is not always immediately possible, but it can be set as a goal to develop a new approach to materials in general, split into "new raw materials" and "secondary raw materials". In an ideal world, all soils and waste can always be reused, and any remediation of contaminated soil would lead directly to the conversion of the contamination into secondary raw materials. We are however still a long way from that, so that an interim solution is needed for this contamination/waste in the meantime. But this should not prevent the legislative framework from already being directed towards that goal and should not prevent ambitious stakeholders from initiating resource recovery and rehabilitation projects.



Figure 16: Process flow outlining an approach to a successful recovery project on PMSD.



6 VALORISATION **OPTIONS**

When it comes to recovering valuable metals from ores or waste materials, there are several techniques available to extract the desired minerals. Two of the most common methods are mechanical and thermal recovery. Mechanical recovery involves physical processes such as crushing, grinding, and screening to separate the target mineral from the waste rock (detailed information in <u>Benchmark report on mineral processing for potential resources extraction for reuse on a PMSD</u>). Thermal recovery, on the other hand, uses high temperatures to drive off impurities or extract metals from the ore (detailed information in <u>Benchmark report on metallurgical processes that are recommended to be applied on PMSD</u>).

Another option for metal recovery is hydrometallurgy, including chemical and bio leaching. Chemical leaching involves the use of chemical solutions to dissolve the target minerals. This technique is particularly useful for low-grade ores or complex ores that cannot be easily processed using mechanical or thermal methods. Chemical leaching can be further divided into techniques such as acid leaching and alkaline leaching, depending on the type of chemical solution used.

Bioleaching is a natural process that uses microorganisms to dissolve the target minerals. This method is particularly useful for ores with low metal concentrations or ores that are difficult to process using traditional methods. Bioleaching is an eco-friendly and cost-effective option, but it requires careful control of the microorganisms and conditions to ensure optimal results.

Overall, the choice of recovery technique depends on a variety of factors, including the type of ore or waste material, the desired minerals, and economic and environmental considerations. By understanding the different options available, it is possible to develop efficient and sustainable recovery strategies that meet the needs of the mining industry and society as a whole.

In addition to raw material recovery, revegetation of PMSD for their renaturation is also important. Plants cultivated on PMSD can potentially accumulate contaminants present in the soil, especially metals, and so when plants are harvested, they become a new waste that may need to be treated. Several options exist to reuse the biomass and among them, the concept of eco-catalysis, which is a new scientific approach that combines ecology and chemistry, is a promising and emerging concept.

It is important to remember that the overall economic assessment of material recovery needs to consider all available resources not just the majority component. The cumulative recovery of multiple components plus the value of the resulting recovered land can make the difference between an economic or profitable recovery or not. It is likely that the processing of materials from a PMSD site will require the utilisation of a number of the techniques described to fulfil its potential and maximise valorisation.



6.1 MECHANICAL RECOVERY TECHNIQUES

Mechanical techniques for recovering metals from soil and waste materials from PMSD involve physical processes that rely on mechanical forces such as crushing, grinding, and screening to separate the metal-bearing materials from other constituents. The most common mechanical techniques used for metal recovery include magnetic separation, eddy current separation, gravity separation, and flotation.

Magnetic separation involves the use of magnetic forces to separate magnetic materials from non-magnetic materials, by means of a magnet that generates a magnetic field and a conveyor belt that carries the metal-bearing material through the magnetic field. The magnetic field attracts and separates the ferrous metals from the non-magnetic materials.

Eddy current separation involves the use of electrical forces to separate non-ferrous metals from non-metallic materials. This process involves creating a rotating magnetic field that induces electrical currents in the metal-bearing material. The induced electrical currents generate a magnetic field that repels the metal from the non-metallic materials.

Gravity separation involves the use of gravity forces to separate materials based on their density. This technique is commonly used for separating heavy metals such as lead, zinc, and copper from lighter materials. In gravity separation, the metal-bearing material is fed onto a sloping surface, and the material is washed with water. The heavy metals settle on the surface, while the lighter materials are washed away.

Flotation is a process that involves the use of air bubbles to separate materials based on their surface properties. This technique is commonly used for separating metals such as copper, lead, and zinc from their ores. In flotation, the metal-bearing material is ground into a fine powder and mixed with water and a flotation agent. The mixture is then aerated, and air bubbles attach to the metal particles, causing them to rise to the surface of the mixture, where they are collected.

The choice of mechanical technique for metal recovery depends on the type of metal and the properties of the matrix. The efficiency of mechanical techniques can also be affected by the size and shape of the metal particles, the presence of impurities, and the moisture content of the matrix. Therefore, it is essential to carefully evaluate the properties of the matrix and choose the appropriate mechanical technique for metal recovery to achieve optimal results.

6.2 THERMAL RECOVERY TECHNIQUES

Thermal techniques for recovering metals from soil and waste materials from metallurgical sites involve the use of high temperatures to separate metals from other constituents. The most common thermal techniques used for metal recovery include pyrometallurgy and hydrometallurgy.



Pyrometallurgy is a process that involves the use of high temperatures to extract metals from ores, concentrates, and waste materials. The process typically involves several steps, including roasting, smelting, and refining. During roasting, the metal-bearing material is heated in the presence of air, causing the material to oxidize and release gases.

The oxidized material is then smelted, a process in which the metal is melted and separated from other constituents. The resulting metal is then refined to remove impurities.

Another thermal technique for metal recovery is incineration, which involves burning the waste material at high temperatures to reduce its volume and destroy any organic contaminants. Incineration is commonly used for treating hazardous waste materials and can be combined with other techniques such as ash separation to recover metals.

There are two areas of pyrometallurgy technology, which are of interest for recovering metals from soil and waste materials from metallurgical sites, namely rotary hearth furnace and electric arc furnace. Rotary hearth furnace technology has been developed commercially to produce a direct reduced iron or hot briquetted iron product that has a worldwide commercial market. In addition, it is also used to recover the volatile metals of zinc and lead in the steel slag. Electric Arc technology has been developed at the commercial scale for processing a broad range of industrial furnace wastes including steelmaking slag.

Another technique is called the plasma arc process, which involves heating the soil or waste material to temperatures of up to 20,000°C using plasma torches. The resulting molten material is then separated into its constituent metals.

Overall, thermal techniques offer several advantages for recovering metals from soil and waste materials from metallurgical sites, including high efficiency, low costs, and the ability to extract a wide range of metals. However, these techniques can also be energy-intensive and can produce emissions that need to be carefully managed to minimize environmental impact.

6.3 HYDROMETALLURGY

Hydrometallurgy is a process that involves the use of liquid solutions to extract metals from ores, concentrates, and waste materials.

6.3.1 Chemical leaching

Chemical leaching is a process that involves the use of chemical solutions to dissolve the target minerals from ores, concentrates, or waste materials (Figure 17). This technique has been used for decades in the mining industry to extract metals such as copper, gold, and silver. However, the use of chemical solvents such as hydrochloric acid, sulfuric acid, or cyanide poses significant environmental and health hazards. Therefore, the development of green solvents for chemical leaching has gained significant attention in recent years.



Green solvents are a class of solvents that are environmentally friendly, non-toxic, and sustainable. These solvents can be derived from renewable resources such as plant extracts, ionic liquids, or deep eutectic solvents. Green solvents can offer several benefits over traditional chemical solvents, including reduced environmental impact, reduced health hazards, and improved metal recovery efficiency. When using chemical leaching with green solvents, there are several criteria that need to be considered for metal recovery from different soil and metallurgical matrices. One of the most important factors is the choice of green solvent used. Different green solvents have varying abilities to dissolve specific metals and work under different environmental conditions. Therefore, choosing the appropriate green solvent for specific soil and metallurgical matrices can significantly enhance the efficiency of chemical leaching.



Figure 17: Decision Tree for hydrometallurgy leaching

The pH level of the solution is another critical factor that needs to be considered. The pH level can affect the solubility of the target metal and can also influence the activity of the green solvent. The presence of other metals or minerals in the matrix can also impact the efficiency of chemical leaching with green solvents.



The temperature and pressure conditions are also critical factors in chemical leaching with green solvents. The optimal temperature range and pressure level depend on the type of green solvent used and the metallurgical matrix. In some cases, it may be necessary to provide additional agitation or mixing to enhance the solubility of the target metal.

Also the processing time needs to be considered in chemical leaching with green solvents. The process can take several hours to several days, depending on the soil or metallurgical matrix and the green solvent used. Therefore, it is essential to balance the processing time with the efficiency of the process and the economic viability of the metal recovery.

In conclusion, chemical leaching with green solvents is a promising and sustainable option for metal recovery from different soil and metallurgical matrices. To ensure optimal results, it is essential to consider critical factors such as the choice of green solvent, the pH level, temperature and pressure conditions, and processing time. By considering these criteria, it is possible to develop effective and sustainable chemical leaching strategies for metal recovery.

6.3.2 Bioleaching

Bioleaching utilizes microorganisms to extract metals from ores, concentrates, or waste materials (metal-bearing-materials, MBM). It has gained significant attention as an eco-friendly and cost-effective alternative to traditional mining and metal recovery methods. However, the efficiency of bioleaching largely depends on several criteria that need to be considered for metal recovery from different soil and metallurgical matrices (Figure 18).

One of the most critical factors in bioleaching is the choice of microorganisms used. Different microorganisms have varying abilities to dissolve specific metals and work under different environmental conditions. For instance, some microorganisms can operate in highly acidic conditions, while others work best under neutral or alkaline conditions. In addition, selection of microorganisms is also important for material target metals. For example, selection of iron and sulphur oxidising microorganisms instead of sulphur oxidiser is recommended for bioleaching of acid in soluble materials (e.g., pyrite, tungstenite). Therefore, choosing the appropriate microorganisms for specific soil and metallurgical matrices can significantly enhance the efficiency of bioleaching (Table 2).

The chemical composition of the soil or metallurgical matrix also needs to be considered. This includes the pH level, the presence of other metals or minerals, and the particle size. The pH level is especially important since it can influence the activity of the microorganisms used in the process. Some metals may also compete with the target metal for the attention of the microorganisms, affecting the overall efficiency of the bioleaching process. The temperature and oxygen levels are also critical factors in bioleaching. The optimal temperature range for mesophilic bacteria, fungi and cyanogenic microorganisms is between 28° C and 35° C, and high oxygen levels (1.5 – 4.0 mg/L dissolved oxygen concentration) are required for the microorganisms to function efficiently (reference [9]). In some cases, it may be necessary to provide additional nutrients to enhance the microbial activity.





Figure 18: Overview of the bioleaching approaches for optimisation before scaling up



Microbial groups	Acidophiles (Figueroa-Estrada et al., 2020; Ma et al., 2021; Rodrigues et al., 2018; Tian et al., 2022)	Fungi (Amiri et al., 2012; Deng and Peng, 2016; Li et al., 2015; Oayyum et al., 2019)	Cyanogens (Arab et al., 2020; Liu, Li and Ge, 2016; Srichandan et al., 2020)
Species	Acidianus brierleyi, A. infernus, Acidimicrobium species, Acidithiobacillus ferrooxidans, A. thiooxidans, A. caldus, Brevibacillus sp., Ferrimicrobium sp., Ferroplasma acidiphilum, F. acidarmanus, Leptospirillum ferrooxidans, L. ferriphilum, Metallosphaera sedula, Sulfobacillus thermosulfidooxidans, S. thermotolerans, S. acidophilus, S. metallicus, S. acidocaldarius, S. solfataricus, S. brierleyi	Aspergillus niger, A. flavus, Penicillium simplicissimum, P. chrysogenum	Bacteria: Pseudomonas fluorescens, P. aeruginosa, P. Caldus, P. putida, P. aeruginosa, Chromobacterium violaceum <u>Fungi:</u> Clytocybe sp., Polysporus sp., Marasmius oreade
Microbial leaching mechanism	thiosulphate pathwaypolysulphide pathway	 acidolysis complexolysis redoxolysis bioaccumulation 	– glycine metabolism
pH range and temperature	 0.8-2.5 Mesophiles: 28-37°C Moderate thermophiles: 40- 60°C Thermophiles: 60-80°C 	- 3.0 - 7.0 - 25-35 ℃	– 7.0-11.0 – 25-35°C
Energy source	 Fe⁺² Reduced inorganic sulphur compounds e.g. S₈, S₂O₃²⁻, H₂S 	– glucose – sucrose	– glycine
Biogenerated leaching agents	– F ⁺³ – H2SO4	 gluconic acid citric acid oxalic acid malic acid 	 hydrogen cyanide

Table 2: Overview of the common microorganisms used for bioleaching applications

The processing time in bioleaching can be relatively slow, taking anywhere from several weeks to several months, depending on the soil or metallurgical matrix and the microorganisms used. Therefore, it is essential to balance the processing time with the efficiency of the process and the economic viability of the metal recovery.

In conclusion, bioleaching is an environmentally friendly and cost-effective option for metal recovery from different soil and metallurgical matrices. To ensure optimal results, it is essential to consider the aforementioned critical factors. Optimizing multiple parameters simultaneously yields a better understanding of the key parameters compared to the one-factor-at-a-time optimization approach. To increase the tolerance of microorganisms to metal toxicity, it is typically recommended to allow for microbial adaptation (acclimatisation).



During bioleaching, automatic monitoring and adjustment of pH is crucial as this parameter greatly influences the process performance. Selecting a microbial group based on the pH value of the material being processed may reduce the consumption of acid and base, thereby lowering costs. By considering these criteria, it is possible to develop effective and sustainable bioleaching strategies for metal recovery.

6.4 ECO-CATALYST PRODUCTION

In addition to raw material recovery, revegetation of PMSD for their renaturation is also important and plants cultivated on PMSD can potentially accumulate metal contaminations. The concept of eco-catalysis production, which is a new scientific approach that combines ecology and chemistry in which metal accumulation in plants becomes an added value, is a promising and emerging concept for PMSD remediation strategies (detailed information in Benchmark report on soil improvement & eco-catalyst production potential on Past Metallurgical Sites and Deposits).

For a revegetation and eco-catalyst production strategy implementation, an important step consists in (i) evaluating the physical and chemical soil characteristics to determine if these soils can support soil vegetation, and, if not, which techniques can improve soil characteristics and (ii) to identify the potential for eco-catalyst synthesis.



Figure 19: Areas potentially interesting for eco-catalyst production on DUFERCO site.



Soil characterisation can be classified in three categories: soil texture, chemical characteristics (linked to soil fertility), and soil contamination.

Soil texture is determined according to the content of clay, silt and sand by using textural triangle and key chemical characteristics to be measured are: pH, cation exchange capacity (CEC), carbonates (CaCO₃), organic carbon content, total nitrogen content, available phosphorus concentration and the electrical conductivity. Metallic trace elements are also to be considered for eco-catalyst production perspective.

For soils improvement, different rehabilitation techniques exist to rehabilitate degraded ecosystems. The traditional method consists of covering the degraded soil with natural materials such as topsoil. However, this technic is very expensive and has a strong impact on the environment linked to the non-renewable resource consumption to which is also added the transport of materials over long distances. Thus, to overcome these disadvantages, other techniques based on introduction of amendments in soils have been developed and are considered for eco-catalyst synthesis perspective. Different kinds of amendments can be used to enhance soil fertility: i) amendments that improve the soil texture, ii) amendments that improve soil fertility (such as fertilisers, liming material, soil improver, and iii) amendments to modify metal availability by decreasing or increasing it.

Sample	Texture	pHwater	CaCO₃	ОМ	Total N	Corg/Ntot	CEC	Available P
	-	-	g kg 1	g kg-1	g kg 1	-	Cmol⁺ kg¹	g kg-1
DFC4	Sandy loam	8.7	72	54	0.72	108	7.5	1.21
DFC5	Silt loam	8.2	27	26	0.35	23	11.9	0.17

Table 3: Example of agronomic parameters of soils collected from DUFERCO site

Table 4: Example of metal concentrations of soils collected from DUFERCO site

Sample	Sb	As	Ва	Cd	Cr	Cu	Hg	Pb	Мо	Ni	Se	Zn
	mg kg ¹ DW											
DFC4	4.1	170	150	8.3	200	140	0.17	820	15	130	3.7	4,100
DFC5	<1	8	90	<0.2	54	16	<0.05	15	0.94	23	1.0	89

Thus, based on historical studies of sites, aerial photos, site visits, and some soils analyses, site specific strategies can be considered for plant cultivation and eco-catalyst production.



Following the soil characterisation, lab tests are then realized to define the best conditions to produce plants (ryegrass *Lolium perenne* L.).



Figure 20: Example of germination essay with soils collected from DUFERCO site

The next step consists of realising greenhouse experiments to evaluate ryegrass development and the transfer of metals from the soils to the vegetative part of ryegrass. Moreover, different amendments are also tested to increase the transfer of metals from soil to plant.



Figure 21: Example of ryegrass development with soils from DUFERCO (a), POMPEY (b) and VIEILLE-MONTAGNE/STPI and (c) eco-catalysts obtained from greenhouse experiments with DUFERCO soils.



After ryegrass development, the ryegrass biomass is evaluated as well as the metal concentrations in the ryegrass shoots. If the metal concentration in ryegrass is considered sufficient, the last step consists in producing eco-catalysts. To obtain eco-catalysts, ryegrass biomass is heated in a muffle furnace and the resulting ashes are treated with HCl aqueous solution. The mixture is stirred, and the final suspension is poured through a filter paper in a Buchner funnel. After cooling, the mixture is filtered through celite, and the filtrate is evaporated to dryness. The resulting solid is dried at 80 °C for 12 h to obtain an eco-catalysts. After eco-catalyst production and according to the metal present in these eco-catalysts, synthesis of molecules can be evaluated and compared to classic catalysts.

6.5 REFERENCES

- Amiri, F., Mousavi, S. M., Yaghmaei, S., & Barati, M. (2012). Bioleaching kinetics of a spent refinery catalyst using Aspergillus niger at optimal conditions. Biochemical Engineering Journal, 67. https://doi.org/10.1016/j.bej.2012.06.011
- 2. Arab, B., Hassanpour, F., Arshadi, M., Yaghmaei, S., & Hamedi, J. (2020). Optimized bioleaching of copper by indigenous cyanogenic bacteria isolated from the landfill of e-waste. Journal of Environmental Management, 261, 110124. https://doi.org/10.1016/J.JENVMAN.2020.110124
- 3. Deng, X., & Peng, F. (2016). Bioleaching remediation on heavy metal polluted soil using Penicillium Chrysogenum. Chinese Journal of Environmental Engineering, 10(11). https://doi.org/10.12030/j.cjee.201603061
- 4. Figueroa-Estrada, J. C., Aguilar-López, R., Rodríguez-Vázquez, R., & Neria-González, M. I. (2020). Bioleaching for the extraction of metals from sulfide ores using a new chemolithoautotrophic bacterium. Hydrometallurgy, 197, 105445. https://doi.org/10.1016/J.HYDROMET.2020.105445
- Li, G., Diao, N., Wang, Y., Du, K., & Zhang, Z. (2015). Bioleaching of copper from tailings by fermentation broths of Aspergillus niger in pellet form. Huagong Xuebao/CIESC Journal, 66(2). https://doi.org/10.11949/j.issn.0438-1157.20141047
- 6. Liu, R., Li, J., & Ge, Z. (2016). Review on Chromobacterium Violaceum for Gold Bioleaching from E-waste. Procedia Environmental Sciences, 31, 947–953. https://doi.org/10.1016/J.PROENV.2016.02.119
- Ma, L., Huang, S., Wu, P., Xiong, J., Wang, H., Liao, H., & Liu, X. (2021). The interaction of acidophiles driving community functional responses to the re-inoculated chalcopyrite bioleaching process. Science of The Total Environment, 798, 149186. https://doi.org/10.1016/J.SCITOTENV.2021.149186
- 8. Qayyum, S., Meng, K., Pervez, S., Nawaz, F., & Peng, C. (2019). Optimization of pH, temperature and carbon source for bioleaching of heavy metals by Aspergillus flavus isolated from contaminated soil. Main Group Metal Chemistry, 42(1), 1–7. https://doi.org/10.1515/mgmc-2018-0038
- 9. Rawlings, D. E., & Johnson, D. B. (2007). Biomining. In Biomining. Springer-Verlag Berlin Heidelberg. Pages 3, 42, 50.
- Rodrigues, M. L. M., Santos, G. H. A., Leôncio, H. C., & Leão, V. A. (2018). Column bioleaching of fluoridecontaining secondary copper sulfide ores: Experiments with Sulfobacillus thermosulfidooxidans. Frontiers in Bioengineering and Biotechnology, 6(FEB). https://doi.org/10.3389/fbioe.2018.00183
- 11. Srichandan, H., Mohapatra, R. K., Singh, P. K., Mishra, S., Parhi, P. K., & Naik, K. (2020). Column bioleaching applications, process development, mechanism, parametric effect and modelling: A review. In Journal of Industrial and Engineering Chemistry (Vol. 90). https://doi.org/10.1016/j.jiec.2020.07.012
- Tian, B., Cui, Y., Qin, Z., Wen, L., Li, Z., Chu, H., & Xin, B. (2022). Indirect bioleaching recovery of valuable metals from electroplating sludge and optimization of various parameters using response surface methodology (RSM). Journal of Environmental Management, 312, 114927. https://doi.org/10.1016/J.JENVMAN.2022.114927



7 CASE STUDIES - PAST METALLURGICAL SITES AND DEPOSITS

7.1 CASE STUDY 1 – DUFERCO SITE AT LA LOUVIÈRE (WALLONIA)

7.1.1 Overview of historical study

DUFERCO – La Louvière site is located in Belgium, in the province of Hainaut, between Le Roeulx and Morlanwelz. The parcel covers more than 120 ha extending to the north of the urban area of La Louvière (Figure 22).



Figure 22: Location of the site in Wallonia Region (source: Maps from sector plan 46/1)

The surface of the site is occupied as follows:

- About 30 hectares are occupied by buildings, mainly the old factory, the old buildings of the FIBO and the steel mill, as well as administrative and office premises;
- About 50 hectares are occupied by storage areas, scrap, slag;
- About 30 % of the surface is occupied by a green or park area (40 ha), located in the south-west of the site.

Since 1853 to until the end of industrial activities at DUFERCO-La Louvière site, the main activity was the manufacturing of iron. The site was undergoing demolition in May 2022. Figure 23 shows all the buildings that have been demolished since the DUFERCO period and the red perimeter presents the area dedicated to the project NWE-REGENERATIS. It can mainly be observed that one of the main buildings of the steel plant has been demolished, the slag mill hall as well as a large part of the buildings of the old plant. It is still planned to demolish some buildings of the steel plant, the area of the agglomeration, the old factory, the brick store of blast furnaces, and several small buildings of the pump room, electrical substations, workshops, etc. More details can be found in a <u>report summarizing historical data</u> of the site.



The topography of the site has been heavily reworked, filled in, and leveled so that its surface is relatively flat. The average altitude of the site is around 120 - 125 meters. Examination of the geological map indicates that the major surface formation on the site is represented by the Yprésian (Tertiary) sandy-clay complex of the Carnière Formation. This formation is characterized by very heterogeneous clay, silt and gravel fractions.



Figure 23: Map of demolished buildings; green: buildings are already demolished; orange: buildings scheduled for demolition in May 2020

In terms of hydrogeology, the following water tables can be found:

- A surface water table in the fills, discontinuous, heterogeneous and periodic;
- An alluvial water table near the Thiriau, in equilibrium with it;
- A water table in the quaternary silts superimposed on the Ypresian aquitard materials;
- A semi-captive and not very exploitable water table in the deep formations of the Houiller. The piezometric level of this deep-water table is expected to be between 20 and 30 meters deep.

The site is not included in the protection perimeter of a sensitive area such as a catchment associated with the public water supply or a protected natural area.

In terms of hydrology, the site is located on the edge of the Canal du Centre and is crossed by a canalized part of the Thiriau du Luc.The stream meanders underground through the site and its course is today only perceptible on the ground by the implementation of a few inspection chambers or access stacks. The watercourse reappears on the surface at the level of the elevator n°1 of the Canal du Centre. This watercourse is exposed to the industrial discharges of the DUFERCO – La Louvière basin and its quality can be qualified as poor. It is estimated that the discharge from the industrial site into the Thiriau represents 60 to 70% of the stream's flow before its confluence with the Haine. The qualitative impact of the industry on this surface water is therefore qualified as significant.



7.1.2 Scanning of the site's revalorization potential: SMART PHOENIX

This section presents the step-by-step approach to apply the REMICRRAM methodology (see chapter 2.2) on DUFERCO-La Louvière site. Figure 24 summarizes each stage and how the expert appraisals required to reach a decision on the recovery potential were carried out.



Figure 24: The NWE-REGENERATIS methodology (REMICRRAM)

7.1.3 Site screening

Based on the REMICRRAM methodology (see chapter 2.2), the site is screened for its potential for metal recovery, mineral extraction, soil enhancement and eco-catalyst production. The methodology relies on data coming from various involved expertise, for historical studies, geophysical investigations, lab and pilot tests, excavation, mineral processing, metallurgical processes, etc.

7.1.3.1 NWE-MESIS

As part of the first phase of the REMICRRAM methodology, the <u>NWE-MESIS</u> (MEtallurgical Sites Inventory Structure (see chapter 2.3) clearly makes the link between the need for standardised data and the REMICRRAM tools.

In practice, in the case of DUFERCO – La Louvière site, filling in the inventory is useful firstly to directly identify important missing information before starting the materials recovery project. Secondly, as the <u>SMART PHOENIX</u> tool is directly included in <u>NWE-MESIS</u>, it allowed to give a first estimation of the recovery potential of the DUFERCO – La Louvière site. All those tools and further results from the site investigation are available on the NWE-REGENERATIS <u>e-library</u>.



7.1.3.2 SMART PHOENIX

Estimating a site's potential for material recovery requires a systematic approach, which starts by answering the 16 <u>SMART PHOENIX</u> questions and assign a confidence level for each answer. These questions are designed to gather general information about the site as well as specifics about the deposits. The figure below shows the 16 SMART PHOENIX answers to the questions concerning the DUFERCO – La Louvière site and the deposit that was investigated during the NWE-REGENERATIS project.



Figure 25: SMART PHOENIX completed with information from the DUFERCO – La Louvière site for the deposit that was investigated in the NWE-REGENERATIS project.

Once the information and confidence levels for the responses have been filled in, the results are obtained in the form of scores, illustrated in Figure 26 for the DUFERCO – La Louvière site. Interested readers will find more information on how to apply the screening tool and how the results and scores for data of a given site are obtained in the <u>SMART PHOENIX guidebook</u>.





Figure 26: Scores obtained after completion of the information for the DUFERCO – La Louvière site for the deposit that was investigated in the NWE-REGENERATIS project

The ranking proved to be excellent for the recovery of metals and minerals (100%), which means it is highly relevant to investigate and characterise the deposit in more detail regarding metal and mineral recovery possibilities and thus move to step 2 of the REMICRRAM methodology. On the other hand, it can be seen that the prima facie estimate of the site's fertility is not that optimal (ranking of only 50 %, with a score just above the threshold value). Since soil fertility improvement and eco-catalyst production are related parameters, a poor estimate of soil fertility implies a prediction of poor potential for eco-catalyst production. Therefore, it will be impossible to achieve maximum potential for eco-catalyst production if the soil fertility estimate is poor. Thus, it can be inferred that the potential of this deposit for eco-catalyst production will not be excellent either, which is indeed the case. Despite the score for eco-catalyst production being above the satisfactory threshold, its ranking is 78 %. All ranking for each material recovery category are graphically displayed.

Globally, the analysis of the total scores for each category of material of the DUFERCO – La Louvière site deposit shows that all scores are sufficient to reasonably assign a favourable diagnosis to further investigate the potential of this deposit for material recovery. It is therefore appropriate to proceed to further site investigation and step 2 of the REMICRRAM methodology: the <u>NWE-SMARTX</u>.



7.1.4 Design and results of geophysical investigations and lab tests

Before proceeding to phase 2 (NWE–SMARTX), in this sub-chapter, we summarize the geophysical investigation of a slag heap from the site of DUFERCO-La Louvière. This sub-chapter will focus on the acquisition strategy, data processing, and geophysical interpretation to illustrate the application of the NWE-REGENERATIS methodology.

The heap (Figure 27 a) is a pile of white slag deposits accumulated from 1994 to 2012 during the deferrization activities of the site. The slag heap is mainly composed of raw materials and by-products of the iron and steel making activities, although heterogeneous waste is likely to be present (e.g., scrap metal, wood, aluminum ingots, refractories, plastics). The geophysical survey objectives were to (i) image the internal structure of the slag, and (ii) identify deposits of interest according to metallic content.

Due to the steep slopes of the deposit, a quasi 3D ERT and IP acquisition (see chapter 3) was planned to provide a dense coverage of the whole white slag heap. It is composed of four 2D profiles containing 64 stainless steel electrodes spaced by 2 m. Data acquisition was carried out simultaneously on combinations of two profiles and inline and crossline measurements were collected to obtain a 3D model of resistivity and chargeability. In the resistivity model, Figure 27 b, the shallower part of the heap appears more resistive than the bottom. Two resistive anomalies are present at the surface in the center of the heap (in yellow). The chargeability model is more heterogeneous (Figure 27 c). Most of the heap is characterized by low chargeability values, inferior to 10 mV/V, with high chargeability anomalies, of up to 250 mV/V. Finally, the resistive anomalies are consistent with low chargeability zones, while the chargeable anomalies are located in average resistivity areas.

In order to interpret these resistivity and chargeability variations, eight pits have been excavated and samples were collected at three different depths (1, 3 and 5 m), to obtain a total of 24 samples. First, laboratory measurements of ERT, IP, and spectral induced polarization (SIP) were carried out. Then, X-ray fluorescence (XRF) analyses were made on the same sample volumes to obtain the chemical composition. These measurements provide more complete information of the geophysical properties of the samples and allow exploring the link between chemical composition and geophysical properties. In the correlation studies, we observed that the Fe is correlated with the chargeability (R = 0.67), while Si is related to low chargeability and high resistivity values. Intermediate concentrations of Fe could be linked to intermediate values of chargeability and a broad range of resistivity values. In most of the samples we observed an intermediate to large concentration of Ca, independently of the Fe concentration. Therefore, we identified three groups of residues with different chemical composition: (1 - red) high Fe concentration; (2 - orange) intermediate Fe and Ca concentrations; and (3 - blue) high Si content, probably due to the presence of inert waste. Finally, we carried out a probabilistic approach to interpret the ERT and IP inverted models through a classification of the field data in terms of the three identified groups. At the end we obtain the 3D distribution of each cluster within the slag heap or a resource distribution model (RDM), see Figure 27 d. More details can be found in the geophysical survey report and correlation report of characterization studies.



This RDM enables to locate waste of interest for potential urban mining or for construction purposes and estimates its potential volume. At DUFERCO – La Louvière site, the volume of the high Fe concentrations materials (group 1 in red) is about 45 000 m³, representing up to 25 % of the investigated slag heap.



Figure 27: 3D view of the white slag heap: (a) orthophotography of the deposit; slice of the 3D (b) resistivity model, (c) chargeability model, and (d) RDM. Small and large gray spheres are the positions of the electrodes of the quasi-3D ERT/IP acquisition and the sampling positions respectively.

7.1.5 Pilot test design and results

The slags found on DUFERCO are a potential alternative material to obtain a new material with enough improved geotechnical and engineering properties to build trafficable platforms for roads. At first, the soil stabilization capabilities of fine slag particles were assessed at a laboratory scale. These initial tests have shown that a formulation incorporating both 0.5 % quicklime and 10 to 15 % of freshly crushed slag of less than 10 mm leads to the improvement of the soil-bearing capacity similar to that obtained through treatment with commercial quicklime introduced at a rate of 2 %. Thanks to these very encouraging results, the implementation of this protocol was applied at a large scale on the DUFERCO site.

A mobile processing unit allowed treating over 3,000 tons of slag material with the purpose to refine the in-situ conditions treatment and to produce three experimental test slabs.

This pilot test took place in two successive phases:



Phase 1: Recovery of reusable materials such as aggregates and fines from slag (March - April 2023)

The treatment process involved preparing the various fractions (fines, aggregates) using mineral processing techniques (crushing, screening, magnetic separation). Firstly, the 3,000 tons of slag were deferrized and screened to obtain two batches of materials with grain sizes of 0-32 mm and +32 mm. The 0-32mm aggregates were further deferrized before passing through a trommel, which separated them into 3 categories of aggregates: 0-10 mm, 10-20 mm and 20-32 mm. Products with a particle size of +32mm were sent to a jaw crusher before being screened to obtain the same particle size fractions as before.



Figure 28: Jaw crusher before screening

All of these materials have been stored on the site pending the completion of phase 2. The table below shows the different masses of the fractions obtained.



Туре	Mass (tons)
Materials subjected to the process	3,047
Ferrous metals	57.69
0/10 screening 1	1484.6
10/20 screening 1	342.5
20/32 screening 1	295.4
0/10 crushed + screening 2	198.5
10/20 crushed + screening 2	56.1
20/32 crushed + screening 2	108.9
Non-crushable materials	32.8
Sludge	34.2
Crushed (maximum crusher setting)	192.9
Loss to ground	243



Phase 2: Demonstration that fines can be used to stabilize soils and as sub-bases for aggregates (June 2023).

In order to do this, three test slabs were built on-site. The two former test slabs refer to soil stabilization using the 0-10 mm fraction and the last one to subbase or subgrade 1 road layers realization using the coarser aggregates fractions (10-20 mm + 20-32 mm).

For the first and second test slabs, 90 m³ of loamy soil have been spread on the ground and compacted over a total area of 225 m² (9 m x 25 m x 0.4 m). Before spreading, the soil was previously mixed and homogenized with binding materials following three different formulations of crushed fine slags (test slab n°1) and screened fine slags (test slab n°2).



Figure 29:The picture shows the two first test slabs before the final testing.

The third test slab concerns road layers obtained by compacting the coarsest fractions. It consists of a 100 m² surface composed of a 0.5 m-thick layer of slag aggregates. This test slab is obtained by spreading and then compacting a suitable mixture of 10-20 mm and 20-32 mm aggregates (screened aggregates + crushed and screened aggregates) to comply with the particle size listed in section 4.4.1. of chapter C of <u>Qualiroutes</u> * (area 3 - tests n°19 to 21). In order to ensure enough bearing capacity and to allow a better compaction, a little amount of the screened 0-10 mm slag fraction was added.

Results

According to Belgian legislation (<u>Qualiroutes</u>), the test reported in the technical standard applicable for road construction in Belgium and which allows to assess the material performance is the plate loading test CME50.01. The plate bearing tests confirm that depending on the considered grain size fraction the slag can be used either for soil stabilization or for road construction sublayers.

Based on those results, it clearly appears that the screened as well as the ground fractions composing the slag stream can be valorized.



7.1.6 Output of DST's: NWE-SMARTX

As high scores in several of the valorization categories were obtained in SMART PHOENIX with a good score for the confidence level, the AI-based decision support tool <u>NWE-SMARTX</u> was applied to the data of DUFERCO – La Louvière site.

With this tool the user can assess the site's valorization potential with more technical details and identify useful processes and methods to be mobilized or investigated for material recovery from the site.

The NWE-SMARTX tool indicates the best valorization processes and treatments to be used for metals and mineral recovery for circular economy opportunities. This is based on decision trees and Machine Learning algorithms related to five expert's domains:

- geophysical techniques for potential resources mapping,
- civil engineering methods for raw material excavation,
- mineral processing processes for mineral recovery,
- metallurgical extraction processes for metal recovery,
- eco-catalyst production potential evaluation.

After downloading and installing the software, the user must fill the input parameters (site specification, samples characteristics, etc.) in the tool. When the user validates the input parameters, the generated outputs indicate for five available modules (related to five expert domains) recommendations or forecasts for further investigations.

Input data (Figure 30) come from investigations and site visits or from laboratory analyses. The values are entered through drop down lists, check boxes or directly into the interface. The results are computed in the *Results* section by hitting the 'Calculate results' button. They are displayed in a dedicated section or module by module as can be seen in Figure 31.

In this case, based on the investigation data and laboratory data, the mineral module recommendations are:

- To valorize the material by using it for CO₂ capture and flue gas desulphurization or as a subbase material in civil work after wet curing to prevent swelling.
- The recommended preprocessing treatments include crushing, screening, and magnetic separation.

In addition to this mineral module, recommendations from the tool concern what geophysical prospection methods are applicable on site, what civil engineering techniques are adapted for excavation, which metal extraction process should be applied on the materials and residues identified on site, what treatment could be used and what could be the potential of using local ryegrass to synthesize bio-based catalysts.



The full results, as presented in the results page of the tool, can be seen in Figure 32:

- For geophysics, the recommended methods are ERT and IP which are the ones that were used on site.
- For excavation, the module recommends performing a stability check and warns the user that drainage and water treatment might be necessary. The cost of these operations should then be taken into account in the business case.
- For the eco-catalyst potential, the module estimates the soil fertility and the expected Zn content in ryegrass. Combining those results the production is not recommended in this case due to limited fertility or its profitability should at least be carefully weighted. Onsite experimentation faced predicted fertility issues and valuable eco-catalyst synthesis.
- For metal valorization, several options are available. See <u>report on pilot scale tests undertaken</u> <u>for metal extraction</u>.
- For the mineral module, several options are available. In the pilot test of the project, the 'wet curing + use as a sub-base material' recommendation was investigated. Results are available in the <u>site specific report on extraction activities performed</u>, pre-processing of bulk samples, raw materials on La Louvière site.

Input Data Results		
erco		
physics Excavation Mineral M	etal Ecocatalyst	
Sync from mineral to metal		
nodulo provider recommendations of pro-treatment an	d options for the minoral fractions valorization of the material w	hich characteristics are introduced as inputs. Global ex
tigated to maximize fraction valorisation.	d options for the mineral nactions valorisation of the material w	nen characterístics are introduceu as inputs, ciobar ev
nvestigation data		
Input information	Value	Unit
Accessibility	Good	۰
Approximative tonnage	150	kt
Hazardness		-
Gross percentage of metal scrap in waste		
Fe/Steel scrap	3,4	%
Al scrap	0	%
Zn scrap	0	%
Pb scrap	0	%
Particle size of waste In-situ		
> 100 mm	80	%
< 100 µm	0	%
aboratory data		
Chemical composition	Value	Unit
Fe grade	22.6	%
Fe° (metal) grade	0	%
Zn grade	0.02	%
Pb grade	0	%
C grade	0	%
S grade	0.2	%
Cu grade	0	%

Figure 30: Input data for the mineral module of the NWE-SMARTX tool for DUFERCO – La Louvière site - slag deposit.



ite Input Data Results	کې ک
uferco	
eophysics Excavation Mineral Metal Ecocatalyst	
Sync from mineral to metal	
his module provides recommendations of pre-treatment and options for the mineral fractions valorisation of the material which ch westigated to maximize fraction valorisation.	aracteristics are introduced as inputs. Global evaluation in combination with the metal module should be
 Investigation data 	
 Laboratory data 	
^ Results	
Name	
Pre-treatment	
Crushing	0
Screening	0
Magnetic separation	0
Technology	
Wet curing + use as sub-base in civil works	θ
CO2 capture and flue gas desulphurization	0
Recommendation	
Valorisation of the mineral fraction of the material is recommended through: Wet curing, CO2 capture The following pre-treatment(s) are recommended: Crushing, Screening, Magnetic separation Note: Pre-treatments thould be applied sequentially. Only one technology should be selected. Several technologies may he Metal scraps are above 1%, valorisation should be considered through particle size classification or gravimetric separation.	wever be applied on various fractions of the materials obtained after pre-treatment to maximize valorisation.

Figure 31: Results of the mineral module of the NWE-SMARTX tool for the DUFERCO – La Louvière site laddle slag deposit

Site Input Data Results				i	• 🄏	P
Duferco						
Geophysics	Excavation	Mineral	Metal	Ecocatalyst		
GRP:Ground penetrating radar (Profiling) 75% 🚯	Drainage and water treatment	Crushing ()	Magnetic separation	Soil Texture	Optimal	0
ERT:Electrical resistivity tomography (Profilir 100%	Stability check	Screening 0	Gravimetric separation (3)	Fertility	Medium	0
IP:Induced polarization (Profiling) 100%	Direct digging	Magnetic separation	Densimetric separation	Zn in plant	161,01mg/kg	0
SRT:Seismic Refraction Tomography (Profilin 50%	Ripping. before digging	Wet curing + use as sub-base in civil works	Eddy current separation (3)			
MASW:Multichannel Analysis of surface wav 50%		CO2 capture and flue gas desulphurization	Electrostatic separation (3)	1		
EMI:Electromagnetic induction (Mapping) 75% (Dewatering (3	1		
Mag:Magnetometry (Mapping) 75% (Sintering and blast furnace (BF)	1		
			Other thermal process (TBRC, BOF,)	1		
			Hydrometallurgy 8	1		
			-			
Recommendation Estimation of values subsq acaptoptics should be possible using the following methods : GPR, ERT, IP, BIL, MAC. When the other state of ERT of TP will require to piece the geomembrane if present. Possibility to do so and budget to repair it should be considered.	Cost and complexity of excavation should be assessed, Particular point of attention are listed in the recommendations below. The implementation of drains is to be evaluated. The inself or a ripper should be determined by pilot tests on site.	Valorisation of the mineral fraction of the material is the following per-trait model of the second second reparting science (1) are recommended reparting science (1) are recommended reparting science (1) are recommended sequentially. Only one technologies multi- nomical technologies multi- mediate (1) are science (1) are science (1) and are science (1) are science (1) and (1) are science (1) are science (1) are science (1) are science (1) are science (1) are science (1) are science (1) are science (1) are science (1) are science (1) are scien	Recommended applicable pre-treatments: Magnetic separation, Eddy current separation, texture operation, Eddy current separation, texture preservice, texture thermal separation of several preventions of the Note: Combination of several preventations and Vote: Combination of Several preventations and the Vote: Combination of Several preventations and the Vote: Several Seve	Prerequisite: potential area fr bare: No building attached or (covered with waterproof cox parking). Fraction of silt clay and sand i for fertility. Solid fertility is dement of the sol initegrated in the business mo Ecocatalyst production might volume should be estimated to in plant (<500mg/kg). Morea production based on other ele Contact expert.	r ecocatalyst must sealed surfaces uting such as roads are within optimal i talyst production m i. The cost should b del. be feasible. Dry bic o compensate for h wer, ecocatalyst sments might be fea	ime or range night xe omass ow Zn asible.

Figure 32: Results of the NWE-SMARTX

More detailed information on how to use the tool can be found by clicking the 'Practical guide' in the software. The procedure to improve NWE-SMARTX Machine Learning algorithms training with end-user additional data can be found in the guidebook for NWE-SMARTX calibration improvement available in the <u>e-library</u>.

Based on those recommendations, several scenarios are applicable for the same site. It's up to the user to choose one of them and then to prepare the cost benefits analyses (phase 3).



7.1.7 Simulation for revalorization potential: Cost-benefit analysis

The third phase of REMICRRAM is the business case development phase. The <u>business case tool</u> takes economic considerations into account and calculates costs and benefits to choose the best valorization scenario based on the recoverable material present on site and site's characteristics. All the necessary information to characterize the deposit are collected, a preliminary assessment of recovery potential and processes has already been made using the SMART PHOENIX and NWE-SMARTX. A resource distribution has already been modelled, as well as any other investigations required for the preliminary work and to design the project (including additional survey). All that remains is to calculate woks cost based on the needs and characteristics of DUFERCO-La Louvière site. The earthworks should thus be designed (e.g. the number of shovels needed, the flowsheet, the stockpile management) and the business case tool shall support you to do that (Figure 33).

The results of the pilot test have been extrapolated to encompass the entire deposit of white ashes, revealing an estimated recovery potential of 381,348 tons of fine and aggregates slag and 8,439 tons of metal scrap available on the site. Additionally, there is an opportunity to backfill approximately 92,895 m³ of void space. These figures demonstrate the significant quantity of recoverable materials present within the deposit.



Figure 33: Earthwork assignment

After calculating the value of the deposit and all the costs for the DUFERCO – La Louvière site's deposits, as shown in Figure 33, the following costs are obtained, summarized in Table 6.

Table 6: Summary	of the	cost-benefit	analysis	for the	DUFERCO	– La Lou	vière site
------------------	--------	--------------	----------	---------	---------	----------	------------

Estimated value of materials [€]	8,753,372
Preliminary studies costs [€]	-115,000
Project studies costs [€]	-145,250
Work costs [€]	-5,413,327
Transportation costs [€]	-1,997,183
Total Net [€]	1,082,612



The valorization of the materials (white ashes for soil stabilization as lime substitute and subbase subgrade embankment) and the void value (backfill) can lead to a significant profit.

The business case for the material recovery project has demonstrated the potential for valuable resource extraction from the identified site. It's a valuable tool that allows the project manager to test different scenarios and adapt it during the design of the project. It makes it easy to compare different scenarios and to make a sensibility analysis of the variation in price of a material. Through careful analysis and evaluation of various factors, including the estimated value of materials, costs of preliminary and project studies, work expenses, and transportation, a site owner gains insights into the financial viability of a potential project.

The estimated value of materials presents a promising opportunity for revenue generation, indicating the presence of recoverable resources within the site. However, it is essential to consider the costs associated with preliminary studies, project studies, works, and transportation. These costs play a crucial role in determining the overall financial outcome of the project.

Despite the expenses incurred during the preliminary and project studies, works, and transportation, the net result of the business case demonstrates a positive value. This suggests the potential for a profitable venture in the material recovery sector.

It is important to note that this business case report provides a preliminary assessment of the financial aspects of the material recovery project. Further detailed analysis and evaluation, including environmental considerations, regulatory compliance, and market dynamics, will be required for a comprehensive understanding of the project's feasibility and long-term sustainability.

With proper planning, effective implementation, and ongoing monitoring, the material recovery project holds the promise of not only generating economic returns but also contributing to environmental sustainability by utilizing valuable resources that would otherwise go to waste.

The business case report serves as a foundation for informed decision-making, enabling stakeholders to evaluate the project's potential, assess risks and benefits, and make well-informed investment decisions. By leveraging the estimated value of materials and carefully managing costs, the material recovery project can pave the way for a sustainable and profitable future.

The business case demonstrates that the material recovery project presents an opportunity to transform a previously underutilized site into a valuable resource hub, contributing to economic growth, environmental stewardship, and a circular economy.



7.1.8 Conclusion and recommendations

In conclusion, applying the REMICRRAM methodology to the DUFERCO-La Louvière site would not have been possible without a large number of expert assessments. Historical studies were used to gather information and results relating to the recovery potential of the deposit and the site. Geophysics was used to determine the volume of the deposit at 235,056 m³ (all categories combined), and to determine the ideal sampling location. All this valuable information was collected in NWE-MESIS and used to implement the entire methodology.

In this way, the scores obtained using the SMART PHOENIX tool (in NWE-MESIS structure) gave a favourable signal for moving on to stage 2 of the methodology. Continued use of NWE-SMARTX made it possible to objectify the recovery potential and provide guidelines for the recovery process. The pilot test resulted in the effective recovery of 2,484 tonnes of fine and aggregate slag and 57 tonnes of metal scraps at the DUFERCO-La Louvière site. This quantity, together with the data from the pilot test and other investigations, extrapolated to the scale of the business cases, led to an estimate of 381,348 tonnes of fine and aggregates slag and 8,439 tonnes of metal scrap that can be recovered on the site. The excellent potential for recovery of the materials means that a net income of \leq 1,082,612 can be estimated for full recovery on the site.



7.2 CASE STUDY 2 – POMPEY (FRANCE)

Pompey is one of the three pilot sites of the NWE-REGENERATIS project. It is a former tailing pond from the iron and steel complex of Pompey-Frouard-Custines, located 10 km North from Nancy. The steel complex was renowned for producing cast iron and special steels, such as ferromanganese (ferro-alloy rich in manganese). The last blast furnace of the Pompey-Frouard-Custines iron and steel complex was stopped in 1986. Over time, a forest ecosystem developed on the former tailing pond.

During the NWE-REGENERATIS project several options for metal valorisation were investigated. Lab scale and when applicable also pilot scale tests were conducted, investigating thermal processes and hydrometallurgy valorisation options on samples concentrated using magnetic separation as pre-treatment. Moreover, several mineral valorisation options were available in a pilot test, investigating the 'wet curing and use as a sub-base material' option with positive results. Two geophysical campaigns, chemical analysis tests, XRF analysis, production of ecocatalyst, and mineral processing were performed on Pompey site. The geophysical and geochemical results were in good agreement and highlighted the presence of four major layers (Lessons learned on the Pompey pilot site).

Altitudes [m]	Group n°	Chemical composition	Geophysical parameter variations	Interpretation
195 - 193.3	4	Scattered	- High rho - Average M - Low MF	Anthropic wastes placed after the closure of the settling pond Present mostly for FP1 and FP4
193.9 - 191.4	1 (cluster B)	Main contributions: Zn, Cu and Pb, Mn	Transition zone: - Decrease of rho - Scattered M - Increase of MF	Settling pond layer n°1
193.2 - 187.4	2	Main contribution: Fe	- Low rho - Scattered M - High MF	Settling pond layer n°2
Very variable limit: ~186.5 for FP1 and FP4 ~189 for FP2 and FP3	3 (cluster A)	Main contributions: Si and K	 Low rho (slightly increasing) Scattered M (decreasing) High MF (slightly decreasing) 	Natural alluvia with high ionic strength electrolyte?

Table 7: Summary of the observations made for each layer of material. The different colours represent the respective layer illustrated in the RAPIDM model for the pilot site of Pompey shown below.



Based on this group classification, a raw distribution model (RAPIDM, see chapter 3.4) for Pompey was generated (<u>Raw materials and pollution distribution models for the 3 PMSD</u>). The results of the field scale probabilistic classification are shown in Figure 34, where the probabilistic classification and the associated probability of occurrence for each group are presented. A high probability of occurrence is represented in red and a low probability in blue. For each cell of each profile, a group number is attributed, corresponding to the maximum probability of occurrence.



Figure 34: Probability of occurrence of the 4 groups estimated using the PCA analysis on geochemical results.

Figure 35 defines the raw materials distribution model for the Pompey site. It was developed using a geostatistical approach combining both the geophysical field datasets and the geochemical laboratory datasets. It allows characterizing the pilot site in terms of layers' extension and thickness, as well as associated chemical composition.

Most of the material corresponds to group 3, which is identified as the natural alluvia in which the settling pond was installed. Two layers corresponding to the settling pond material were identified (group n°1 and 2). Group n°4 corresponds to a layer of anthropogenic wastes posterior to the closure of the steel complex. Their localization in Figure 35 corresponds to topographic heights.





Figure 35: RAPIDM model for the pilot site of Pompey.

Laboratory mineral processing was conducted on two samples from the Pompey pilot site. Although these samples contained high concentrations of several metals (e.g. Fe, Mn, Zn, Pb), their grades are too low, to this day, to justify any economic interest in recovering them using mineral processing techniques. Greenhouse experiments on the production of eco-catalysts were also conducted on 4 samples of the pilot site (Figure 36). The four samples present high zinc concentrations, and good fertility parameters. The samples allow for a very high development rate of the planted ryegrass. Two eco-catalysts were produced from the ryegrass harvest, both highly enriched with Zn (Figure 36 b, c). Thus, the material on site seems to be more suitable for recovery by eco-catalysis than mineral processing.



Figure 36: a) Ryegrass development on samples from Pompey; b) eco-catalyst produced for sample 11-A; c) eco-catalyst produced from sample 12-A.



7.3 CASE STUDY 3 – TEESSIDE (UNITED KINGDOM)

The Teesside site is located in North East England (Figure 37) and is the site of a former integrated steelworks, which handled the entire steel production process from raw materials to finished products. The site is a large one (1.500 ha) with a 160-year history of iron and steel production and the processing of finished products. It comprises large areas of Redcar, Lackenby, Grangetown and South Bank to the South of the river Tees.



Figure 37: Location of Teesside site.

7.3.1 Historical study

As an integrated steelworks, which processed from raw materials to finished products, there were several known areas used for the storage of waste products dating back as far as the 1900s. The site has been used, at varying periods of time, for the storage of feedstock, products, by-products and waste streams. Over the years, due to changes in ownership, regulatory controls and economic conditions, the materials have co-mingled with poor associated recording of the inventories of quantity and quality of materials.

A huge amount of historical data was found to exist and a <u>desktop review of historical records</u> of site activities was carried out. Since the land area is so large and diverse, it was important to maximise available knowledge to provide contextual guidance for the recommended locations of geophysical surveying and sampling.



7.3.2 Identification of test sites

A number of areas were identified for consideration, all of which have been used for waste management. These are shown in Figure 38.



Figure 38: Waste management facilities within PMSD pilot site Teesside.

Initially, the South Lackenby Effluent Management System or SLEMS was identified for site investigation. An area of 22 hectares, the SLEMS was used for waste handling and treatment for Basic Oxygen Steelmaking (BOS) oxide waste. Initial sampling was done and laboratory tests performed to assess the feasibility of recovering valuable metal content from it.

After samples were taken for laboratory analysis and during the preparation for geophysical examination, the SLEMS landfill was repurposed for work involving land development at Teesside. Although this was a temporary use, the time scales were such that it was not feasible to perform any site work at this location during the project.

An alternative area for geophysical study was sought within Teesside. Following site visits to identify potential areas, it was agreed that the CLE31 landfill would be a suitable location (see Figure 38 and Figure 39).





Figure 39: CLE31 landfill site.

CLE31 is a closed waste disposal site and was used primarily for the disposal of blast furnace and steelmaking slag with a small percentage of general site waste. The site was used from the 1930s until it was closed in 2002.

7.3.3 Sampling

Extensive historical data regarding the SLEMS site is available from previous on-site investigations. The ground was reported as comprising BOS oxide in the form of a slightly, gravelly silt underlain with slag, refractory bricks and other wastes. The average BOS oxide content found was 63 % and 37 % slag.

Samples were collected and distributed between The Materials Processing Institute, Cranfield University and CTP. They performed various types of testing, such as chemical analysis, material separation testing, pyrometallurgical testing, and hydrometallurgy.



7.3.4 Geophysical survey

A <u>geophysics campaign</u> using several techniques was carried out on the CLE31 landfill site.

- Topography measurement
- Geomagnetic surveys
- Measurements of the geomagnetic field amplitude
- Measurements of surficial magnetic susceptibility
- Electro Magnetic Induction (EMI) survey (Figure 40)
- Electrical Resistivity and Induced Polarization (IP) tomography profiles

The geophysical survey carried out on CLE31 shows that the material filling the heap has variable and heterogeneous geophysical properties. Magnetic and electromagnetic maps show randomly distributed parameters. The magnetic and electromagnetic surveys are affected by the local variations of magnetic susceptibility which is high and highly variable on these kinds of wastes from iron metallurgy. Thus, magnetic and electromagnetic surveys are difficult to interpret in such context and these techniques are not recommended for mapping wastes from iron metallurgy. However, they highlighted the heterogeneous nature of the wastes that are likely to have been deposited randomly.



Figure 40: ElectroMagnetic Induction (EMI) DUALEM-421S equipment

The electrical resistivity and IP tomography are not affected by highly magnetically susceptible materials. These techniques allowed distinguishing a layer as well as several anomalies with homogeneous electrical properties within the heap. The slag heap was likely built from waste of very different origins.



As electrical resistivity and IP tomography techniques are not sensitive to magnetic susceptibility, it is possible to classify the slag and other metallurgical wastes on Teesside site (CLE31) based on their resistivity and IP signatures. At least, 3 different types of slags have been identified. Using results from the 5 ERT profiles carried out on the slag heap, the geometry and the volume of these different kinds of wastes can be calculated. An example of the ERT results can be seen in Figure 5.



Figure 41: Electrical resistivity tomography - ERT2. Top: Electrical resistivity cross-section; middle: Chargeability cross-section; bottom: Metal Factor cross-section.

To support the geophysics results and aid in the interpretation five specific boreholes were proposed to further characterize geophysical signatures in terms of metallic element concentrations, metallic particle identification, and granulometry.


However, it was not possible to develop a quantitative estimation of the targeted volumes, since deeper sampling on site was not achieved during the project. Due to the heterogeneous nature of the deposits observed by the geophysical tools, a quantitative interpretation is only possible using targeted samples that are analysed at laboratory scale, both for geochemical concentration estimations of chemical elements and geophysical properties.

The most useful geophysical methods at the Teesside site were found to be ERT and IP. They allowed for volume estimations of revalorization of materials and the detection of various interfaces and layers within the deposits. In conclusion, the geophysical survey emphasized the heterogeneous nature of the waste materials on the Teesside site and stressed the importance of complementary analyses for understanding and revalorizing the deposited slags.

7.3.5 Laboratory tests and analysis on valorisation options

On Teesside site the metal recovery options were investigated. The choice of recovery technique depends on a variety of factors, including the type of ore or waste material, the desired metals, and economic and environmental considerations. By understanding the different options available, it is possible to develop efficient and sustainable recovery strategies. More details on valorisation options can be found in chapter 6.

7.3.5.1 Chemical analysis

Chemical analysis carried out by all partners confirmed that the materials collected from the SLEMS area was very high in Iron, accounting for more than 50 % of the samples.

7.3.5.2 Mineral separation

<u>Mineral separation</u> was carried out at CTP in Belgium and at Buntings Magnetics in the UK. Separation tests using mineral processing techniques including wet screening, magnetic separation and electrostatic separation were carried out on a lab scale. The chemical analysis of the separated fractions showed that the screening did not allow for a separation of a richer ferrous fraction as each size range had a very similar chemical composition (55-57% Fe). In addition, the material was found to be predominantly magnetic. The performance of magnetic separation led to the removal of most of the material at very low magnetic susceptibilities. Magnetic separation techniques did not seem efficient for SLEMS BOS oxide material, clearly indicating that the magnetic fraction (iron oxide) was fully distributed through the material and in every size fraction. There was little or no enrichment of the materials by screening, electrostatic or magnetic separation.



7.3.5.3 Pyrometallurgy

Pyrometallurgy tests explored the efficiency of extracting useful metallic iron from the SLEMS area. A carbothermal reduction was employed at high temperature to reduce the iron oxide compositions into metallic Fe in an attempt to valorise these materials. The SLEMS samples were melted in an induction furnace of 45 kW (Figure 42) to recover the constituent metals present in the samples. The samples were mixed with graphite powder, which acts as a reducing agent.



Figure 42: Experimental setup for melting and schematic of the induction furnace system.

Initial trials showed poor recovery, because the material failed to melt completely even at elevated temperatures. The chemistry of the SLEMS material was adjusted by the addition of silica, which reduced the basicity of the slag reducing the melting temperature. This was intended to maximise the iron recovery while reducing the necessary furnace temperature in order to obtain maximum yield at minimum temperature.

Despite the attempts at modification of the chemistry, the only moderately successful attempt was when a high temperature of 1700 °C was used and then only approximately 88 % of the iron was recovered. Thus, it was concluded that the SLEMS material, which has very high iron content, is of high value. However, the yield of metallic iron was low, which makes the pyrometallurgical recovery using these techniques, not a feasible option.



The process used was a one-step melting process. It may be possible to separate the reduced iron from the remaining slag using either a secondary melting process or a crushing and magnetic separation. The material may be recoverable, using a more conventional blast furnace route for example, but this is made difficult by the residual Zinc content. It may be that a secondary process may be able to reduce the Zinc to acceptable levels such as hydrometallurgy.

7.3.5.4 Hydrometallurgy

Samples taken from Teesside were sent to Cranfield University and subjected to hydrometallurgical testing. <u>Hydrometallurgy</u> is a process by which desirable elements are extracted by leaching them from a material using <u>chemical solvents</u> or <u>biological agents</u>. The samples were subjected to batch and column testing to test for the recovery of various metals, such as Y, Ce, Nd, Li, Co, Cu, Zn, Mn, and Al. The impact of different irrigation rates on bioleaching rates, comparing column-scale experiments (Figure 43) with shake flask experiments and control were explored.



Figure 43: Column setup for chemical extraction trials

Using chemical leaching, a column study was conducted with green solvents. The study investigated the effect of continuation infiltration and recirculation on solvent extraction efficiency compared to agitation and settlement. Results from the study show varying total metal extraction efficiencies for different solvents.



Bioleaching and chemical extraction efficiencies for selected metals were compared by leachate analysis using ICP-MS. Bioleaching outperformed chemical extraction for certain metals, while chemical extraction was comparable for others, indicating opportunities for selective metal recovery in sequential processes. Different solvents have been shown to perform differently for different target elements. The results from these static and dynamic studies have been used to develop an initial criterion for green solvent selection. It has been suggested that a sequential approach where different solvents are applied in order needs to be used for the most effective elemental extraction.

7.3.5.5 Conclusion

The Teesside site contains a significant volume of potentially valuable waste material currently stored in a landfill on the PMSD site.

Geophysics investigations have shown that it is possible to map existing deposits, which when combined with suitable small-scale sampling, could lead to an estimation of potential material for recovery and valorisation.

Laboratory work has shown that there is potential for recovery of material by a combination of techniques including hydro- and pyro- metallurgy.

The excavation of steel waste from the PMSD will require a well thought out approach that considers safety, efficiency, environmental protection, and regulatory compliance. Engaging experienced contractors and environmental experts is essential to develop and implement a successful excavation plan. By employing the right techniques and equipment, the valuable steel waste can be efficiently and safely removed from the PMSD, while minimizing its impact on the environment.



SUMMARY





CONTACT PERSONS

Feel free to contact one of the project partners below or check out the <u>project website of NWE-</u> <u>REGENERATIS.</u>

Lead partner :

BELGIUM	Marta Popova (SPAQuE)	m.popova@spaque.be
	Avenue M. Destenay 13	
	4000 Liège, Belgium	

Contact details of the project partners :

BELGIUM	Renaud De Rijdt (ATRASOL)	<u>renaud.derijdt@atrasol.eu</u>
	Ugo Falcinelli (DUFERCO)	u.falcinelli@duferco.be
	Antoine Masse (CTP)	antoine.masse@ctp.be
	Frédéric Nguyen (ULiège)	f.nguyen@ulg.ac.be
	Eddy Wille (OVAM)	ewille@ovam.be
France	Tristan Debuigne (IXSANE)	tristan.debuigne@ixsane.com
	Pauline Kessouri (BRGM)	<u>p.kessouri@brgm.fr</u>
	Jérémie Renault (TEAM2)	<u>ext-j.renault@team2.fr</u>
	Christophe Waterlot (JUNIA)	christophe.waterlot@junia.com
GERMANY	Pascal Beese-Vasbender (BAV)	<u>pbv@bavmail.de</u>
	Christian Wolf (TH Köln)	christian.wolf@th-koeln.de
THE UNITED KINGDOM	Alan Scholes (MPI)	alan.scholes@mpiuk.com
	Frederic Coulon (CU)	f.coulon@cranfield.ac.uk



FUNDING

Interreg North-West Europe Cooperation Programme funded by the European Commission European Regional Development Fund (ERDF) Interreg-NWE Programme Priority 3: Resource and materials efficiency Specific objective: To optimise (re)use of material and natural resources in NWE

NWE-REGENERATIS Funding:

Total budget received from Interreg North-West Europe: € 4.23 million of ERDF

Total Project Budget: € 7.06 million







