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D.T3.1.3 Performance report on new developed Geophysical Characterization Method (GCM)

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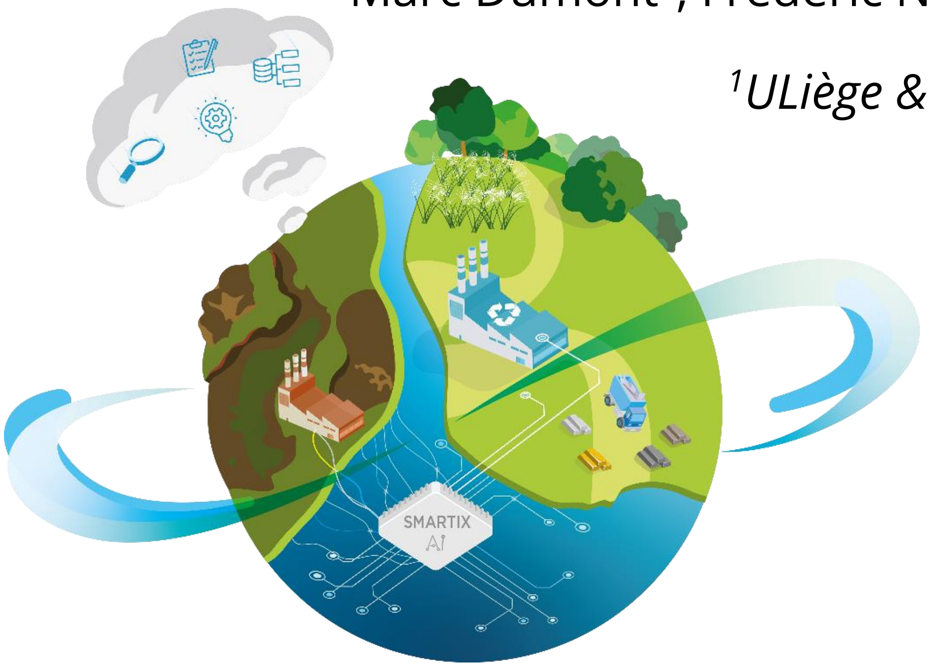

SUMMARY

1 INTRODUCTION

During the NWE-REGENERATIS project, ULiege and BRGM conjointly developed a new geophysical approach to characterize past metallurgical sites and deposits (PMSD). The main objective is to build a raw materials and pollution distribution model (RAPIDM) of the site. Here, we first summarize the general methodology applied and adapted in the different sites of the project. Then, we analyse the pros and cons of the geophysical methods used in the field and in the laboratory for the different types of sites and materials. Finally, we present the experiences gained and recommend the most suitable methods to characterize a given PMSD.

2 INTEGRATED METHODOLOGY FOR GEOPHYSICAL INVESTIGATIONS IN PMSD

The NWE-REGENERATIS methodology aims to carry out a cost-effective and non-invasive geophysical characterization of PMSD, integrating ground truth data from optimized sampling and laboratory measurements. The methodology is composed of five steps shown in Figure 1.

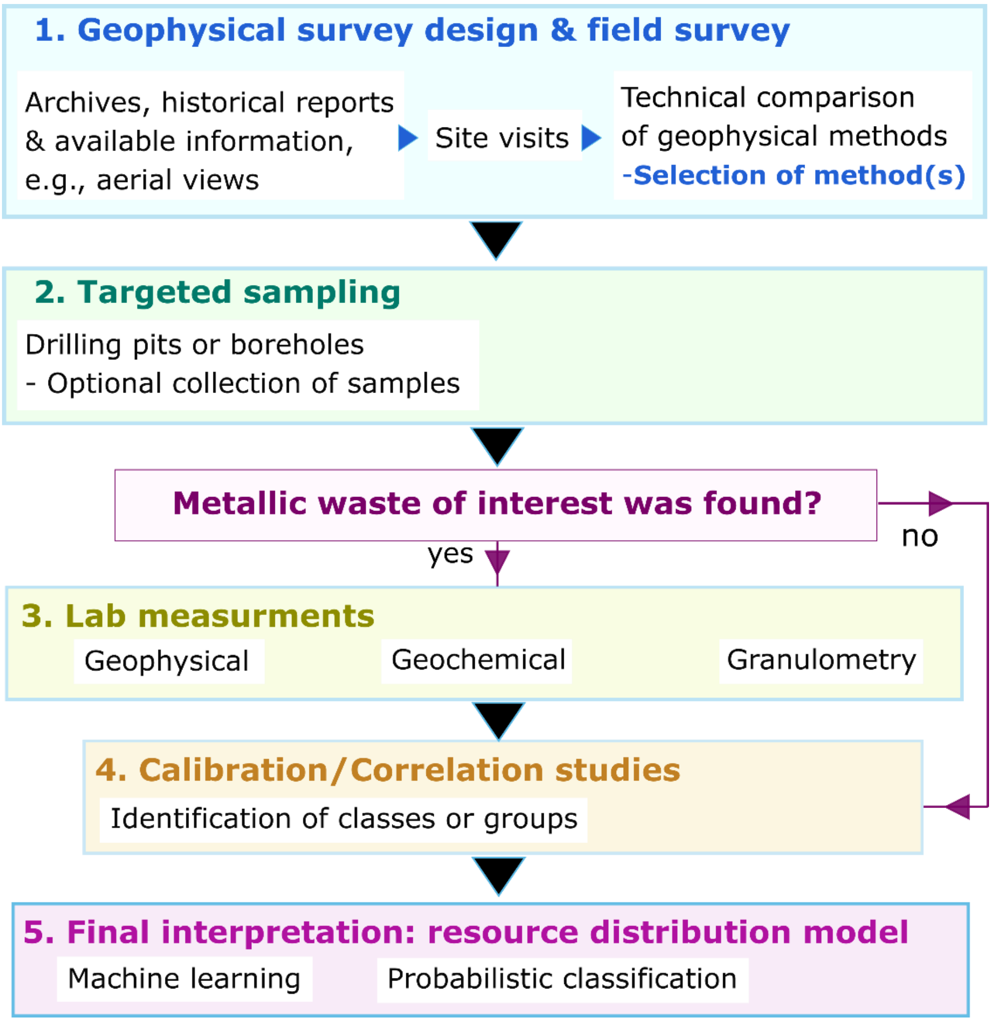

Figure 1 : General methodology of the Geophysical Characterization Method in PMSD.

The first step is to conduct a geophysical survey in the field using the most suitable methods. The design of the survey is based on: 1) historical archives and inventories of the site as well as available online information and 2) current physical situation, i.e., vegetation, topography, accessibility, etc. This information could be then used to answer the decision tree or SMARTIX tool defined during the project in order to select the more appropriate geophysical methods. Detailed information is described in the decision tree reported in DI3.2.4.

In the second step, we design a targeted sampling based on the results obtained in the field, i.e., selective excavations of boreholes/pits at locations where strong contrasts or anomalous values of a physical property are found and collection of samples at depths of interest. The sampling survey is design to provide geochemical analysis of area with contrasted geophysical properties. Then, the laboratory analysis will provide meaningful information of the geophysical contrast imaged in the field.

In the step three, chemical/mineralogical analyses are carried out in the laboratory on the samples collected. The first objective is to provide geochemical analysis of slag with different geophysical properties. In addition, geophysical measurements can be done on the samples in order to avoid any scale effects. If no geophysical laboratory measurements are done, geophysical properties collocated with the samples will be used afterwards.

During the step four, we study the correlations between the geophysical lab measurements and the chemical/mineralogical analyses. A first step could consist in identifying linear relationships or clusters/groups of samples with different chemical compositions and geophysical properties that may constitute a training dataset. Using these correlations analysis, we develop a raw materials and pollution distribution model from the field geophysical data (RAPIDM) . To this aim, a probabilistic approach may be used (Isunza Manrique et al. 2019; Hermans and Irving 2017), an algorithm of machine learning (Inauen et al. 2020; Bressan et al. 2020; Lysdahl et al. 2022) or other non-supervised learning methods (M. Dumont et al. 2018; Sabor et al. 2021; Whiteley et al. 2021).

Note that each step of the NWE-REGENERATIS geophysical methodology has to be adapted according to the site-specific conditions. The types of PMSD, the materials that can be found and the applicability of certain geophysical methods in the field vary from site to site. Limitations related to topography, the infrastructures present on site and budget may affect the sampling survey. The methods to be used in the laboratory are those which proved to be suitable in the field, therefore they may vary for each site. Then, the type of chemical analysis can also be different for the samples of different sites depending on type and quantity of material, budget, time-limitations, etc. Finally, several supervised and non-supervised learning algorithms can be applied to derive a RAPIDM, depending on the availability of data for interpretation. In the following sections we present the different application used on NWE-REGENERATIS sites highlighting the strength and limits in order to analyse the performance of the geophysical approaches.

3 PERFORMANCE OF THE INTEGRATED METHODOLOGY

3.1 PILOT SITES

3.1.1 First pilot site: Teeside (UK)

3.1.1.1 Geophysical survey in the field

The geophysical survey in Teesside was conducted over the CLE31 sites. This slag heap is composed by waste from iron factories. The survey was composed of: (i) a photogrammetry survey using a drone; (ii) electromagnetic induction (EMI), magnetic susceptibility and magnetic mapping; (iii) electrical resistivity and induced polarization tomographies, called ERT and IP respectively.

Obstacles

The historical data on the specific slag heap are very sparse, making the design of the geophysical survey less accurate.

Moreover, the site is highly heterogeneous with a wild variety of metallurgical deposits highlighted by the presence of metallic scraps and other metallic waste at the surface. This heterogeneity is complicated to characterize with geophysical methods (see deliverable DI1.2.4).

EMI mapping is particularly impacted by the heterogeneous nature of the waste: the EM results are noisy, and don't allow any quantitative interpretation.

The magnetic field gradient mapping is also impacted by the heterogeneous nature of the slag heap. Indeed, no trends were identified on the map, and only large local variations are visible on the interpolated map. Only qualitative information on the variability of the wastes magnetic nature can be extracted.

Regarding the surficial magnetic susceptibility map, three homogeneous areas can be identified, including a central area, with developed vegetation. Quantitative lateral interpretation in terms of areas could be extracted from the data. However, because the methos only investigates the first 5 cm of the deposit, it cannot be used for volume estimations.

Opportunities and recommendations

The 2D ERT/IP profiles have proven their efficiency no matter the heterogeneity of the slag heap. The imagery provided by these methods is reliable and has been used to define the targeted sampling survey.

The development of UAV survey in the last months of the project open new perspectives. As developed bellow (subpart 3.2.4), different devices can be mounted on UAV to provide a dense continuous mapping in the heap, e.g., multispectral measurements.

3.1.1.2 Targeted sampling

Obstacles

Using geophysical imagery, especially ERT/IP profiles, a targeted sampling survey has been designed (see deliverable DI1.2.1). However, due to environmental restrictions and time limits, no sampling approach has been possible during the project.

Opportunities and recommendations

Because of the heterogeneous nature of the investigated slag heap, it is all the more important to collect ground-truth geochemical data. No volume estimation of potential layers to revalorize can be made without these results.

3.1.1.3 Final interpretation: RAPIDM

As the targeted sampling could not be conducted in the site, the RAPIDM was developed following an unsupervised learning approach. The interpretation of the inverted resistivity and chargeability models was based on a clustering algorithm, where an unsupervised identification of groups is created. These groups that present a variation in geophysical properties could be indicators of a variation in geochemical properties as well. Therefore, the RAPIDM model is created under this assumption.

Obstacles

The main obstacle in the creation of the RAPIDM model was the lack of ground truth data, i.e., excavation of trial pits or boreholes and sampling, to calibrate the geophysical data with. This was mostly due to logistic and environmental reasons within the management of the site.

Opportunities and recommendations

In sites under the conditions presented here, we find that unsupervised learning can be an alternative to develop a more quantitative interpretation of geophysical data trough the generation of a RAPIDM. In addition, there are several algorithms that can be used to carry out clustering approaches that can be compared. Lastly, the resulting model can be assessed and validated afterwards if subsequent excavations and sampling may take place.

3.1.2 Second pilot site: Pompey (FR)

3.1.2.1 Geophysical survey in the field

The geophysical survey in Pompey focused on former tailing ponds where by-products of iron-based alloys were dumped in the past. The survey was made in two distinct steps. In the initial step, a rapid exploration was conducted, involving the use of a single electrical profile and punctual magnetic susceptibility measurements at various depths within an existing trench. Additionally, an initial sample collection was performed. This preliminary investigation allowed for a preliminary assessment of the geophysical properties of the site and its composition.

The second step of the survey was conducted a few months later and consisted in a more comprehensive characterization of the site (see deliverable DI2.2.1). It included the coverage of the area of interest with electromagnetic induction (EMI) and magnetic methods followed by 5 electrical resistivity tomography (ERT) and induced polarization (IP) profiles. They were complemented with 3 active seismic profiles (multi-channel analysis of surface waves - MASW) along the same profiles than 3 of the ERT lines.

Obstacles

The geophysical survey was complicated by the dense vegetation present on the site, which prevented proper coverage by electromagnetic and magnetic methods and slowed down the deployment of electrical and seismic profiles. Electromagnetic and magnetic data were too sparse to provide any useful additional information compared with other deployed methods.

Opportunities and recommendations

The combination of ERT and IP data provided the most valuable information for determining the internal structure of the settling ponds, as well as the potentially more metal-rich zones. The fine material composing the deposits allowed the MASW results to confirm the vertical extent of the deposits identified with the ERT and IP.. A complementary approach that could add value to the ERT and IP results, is the use of frequency information for both the electrical conductivity and chargeability. This can be done through SIP measurements in the field (with a different instrument), or through new measurement modes with the IP instrument used in the field in Pompey. Tests are undergoing. This additional method would help interpret the deposits in terms of textural parameters, as well as hydrogeological properties, and metallic particles characteristics.

3.1.2.2 Targeted sampling

Based on the results of the field geophysical survey (ERT and IP),, four sampling locations were suggested.

Obstacles

The Pompey soils are delicate to excavate and sensitive to water, leading to poor mechanical properties at the surface. Because of the nature of the deposits, no heavy machinery is allowed to enter the site. The dense vegetation makes the site even less usable for heavy machinery. A lightweight auger was thus used for the sampling.

Opportunities and recommendations

Samples were collected all the way until the bottom of the settling pond was detected visually while collecting the samples. The depth of the deposits estimated using geophysical investigations could thus be double-checked through the geochemical lab measurements.

The lightweight auger used had a helicoidal structure, making it impossible to preserve the structure of the samples. A recommendation for future work to preserve the structure of the sample would be to use a core drill and preserve the core samples extracted in good conditions (temperature and humidity) in order to extract even more data in the laboratory experiments.

3.1.2.3 Laboratory measurements

45 samples were taken by BRGM using a light core drill for depths ranging between 0 and 9.2 m at 4 different locations. Geochemical and geophysical laboratory measurements were planned on these samples. For the geochemical measurements, a portable XRF tool (p-XRF) was used on dried and grinned samples (see deliverable DI2.2.2).

Obstacles

Because the sample volume was small (around 0.1 dm^3), no existing sample holders at BRGM could accommodate the Pompey samples. No budget and time was allocated to the development of a new sample holder within the scope of the project. Attempts have however been made, without reaching satisfying results.

Opportunities and recommendations

The p-XRF results could be used to draw correlations with the geophysical field results. The limit between the PMSD deposit layers and the natural alluvia was detected clearly.

Only simple geochemical analysis was led during the project, allowing detection of chemical element concentration variations. To go further in the identification of the nature of the metallic particles, it would be interesting to run other geochemical analysis such as X-ray diffraction (XRD) and inductively coupled plasma (ICP), to understand their mineralogy.

3.1.2.4 Correlation studies

Two different statistical analysis were used to identify correlations between: (1) the chemical elements, and (2) the chemical elements and the geophysical field parameters:

- The Pearson's correlation coefficient analysis that allows correlating variables 2-by-2
- The principal component analysis (PCA) that reduces the number of dimensions considered in the correlation analysis.

Obstacles

The Pearson's correlation coefficient analysis is interesting because it shows positive and negative correlations, but it is not very well suited to analyse datasets with a lot of different variables (31 chemical elements in our case, or 34 parameters if we add the geophysical parameters).

As we were not successful with the geophysical laboratory measurements, we used field geophysical measurements from inverted profiles close to the location of the samples extracted. Comparing measurements taken at 2 different scales, and not exactly at the same location, targeting the same volume makes the comparison more uncertain.

Opportunities and recommendations

Using PCA analysis on the chemical elements, we were able to distinguish clusters with different dominant chemical components, and identified depth. These clusters were used to build the RAPIDM model for the Pompey site.

3.1.2.5 Final interpretation: RAPIDM

The interpretation of the six ERT/ IP inverted profiles was done in terms of the clusters of different chemical compositions distinguished in the previous step. A probabilistic approach was developed to identify the clusters' areas on each profile using the following data: resistivity and chargeability (inverted) values at the position at which the boreholes were drilled, and the position {*x*, *y*, *z*} of the samples where the geochemical dataset was established. The joint conditional probabilities of each group in the whole acquisition domain were then estimated. The cluster with the maximum joint probability was associate to each cell of the profiles (see deliverable DT3.1.1).

Obstacles

One of the main obstacles is that there are only 4 locations for the boreholes, that are not directly located on the electrical profiles. The correlation between the geochemical and field geophysical dataset is thus hard to establish.

Moreover, the electrical profiles were only inverted in 2D. Estimation of volumes associated to each cluster thus requires interpolation of the results over the entire studied area. 3D acquisition would be recommended for future investigations.

Opportunities and recommendations

The probabilistic approach was suitable to perform a classification of the geophysical data acquired on the Pompey field site, including associated conditional probabilities. Further data treatment is needed to estimate quantitative volumes for each clusters.

3.1.3 Third pilot site : Duferco La Louvière (BE)

3.1.3.1 Geophysical survey in the field

The geophysical investigation carried out in the field prioritized accessible areas of larger potential for resource recovery. Therefore, in this report we focus on the investigations of the slag heap, mainly composed of raw materials and by-products from iron and steel making activities. After the analysis of the historical archives and available information, as well as a couple of site visits we used the following methods: ERT, IP, magnetometry and the two seismic methods, i.e., MASW and seismic refraction tomography (SRT).

Obstacles

There are three main obstacles found during the data acquisition and data processing. First, the measurements with the magnetometer were saturated probably due to the presence of metallic scraps and rebars. Then, the heavy rain, the stability of the heap slopes and the abundant concrete blocks complicated the acquisition and data processing of the MASW and SRT methods. Concrete blocks and other inert waste acted as scatterers for the seismic waves, and therefore complicated the processing and interpretation.

Opportunities and recommendations

The use of the EMI and the ground penetrating radar (GPR) could have helped to resolve the shallowest layers of the slag heap as the subsurface did not present notably large conductivity values. Overall, ERT and IP were the most useful methods to characterize the slag heap and resolve areas of different chemical composition. Another viable approach that could have yielded additional valuable insights is the Spectral Induced Polarization (SIP) method. This particular technique is capable of capturing a wider range of information due to its sensitivity towards textural parameters, as well as geochemical and hydrogeological properties.

3.1.3.2 Targeted sampling

The targeted sampling was designed from the inverted 3D models of ERT and IP, where several lateral and vertical contrasts were observed. Samples were collected at depths between 1 and 5 meters at 8 locations of the heap.

Obstacles

The hardness of the slags at some locations and the stability of the slopes limited the excavations at certain locations and at depths larger than around 6 m. Additionally, the budget did not integrate the use of a more specialized drilling technology for hard soils. On this basis, the interpretation of the 3D ERT and IP inverted models for depths larger than 6 m could not be validated with ground truth data.

Opportunities and recommendations

The collection of samples at depth intervals of 2 m may be enough to differentiate materials of different chemical composition. In addition the excavation of pits represented a suitable technique that enabled the collection of a large quantity of material per sample. Finally, it is also highly recommended to store the collected samples in sealed containers and to preserve the material in cold environments to keep the samples in conditions as close as possible to those prevailing in the field.

3.1.3.3 Laboratory measurements

For laboratory geophysical measurements, we applied ERT, IP and SIP methods using 1.5 dm3 columns. We studied the impact of water saturation on the electrical properties of the materials tested. To this end, after an initial measurement under field saturation conditions, we saturated the columns with tap water and measured the electrical properties 1) immediately after saturation and 2) after a certain time ($60 < T < 1100$ min).

When the geophysical measurements were finalized, the samples were sent for chemical analyses and particle size distribution determination. First, the samples were analysed using the semi-quantitative method of X-ray fluorescence (XRF). A subset of the samples was then sent for quantitative analysis using X-ray diffraction (XRD) and inductively coupled plasma (ICP).

Obstacles

The saturation of samples using tap water may have affected their composition. In terms of the elemental composition, the mass fraction of the samples that could be effectively characterized quantitatively only represent 50%. The remaining fraction of material corresponds to an amorphous phase with an unknown composition.

Opportunities and recommendations

ERT, IP and specially SIP are promising methods to investigate metallurgical wastes of different chemical compositions. Additionally, employing large columns that replicate the heterogeneity observed in the field has proven to be advantageous. If the effect of different water content is to be studied, it is advisable to use de-ionized water rather than tap water and to wait until the physical parameters (pH, T°, conductivity) of the water flowing from the columns have stabilized before measuring the electrical properties (Placencia-Gómez et al. 2015). It has also been recommended to use water with properties as close as possible to the water on site: either directly collecting water on site (and carry out chemical analysis) or using de-ionized water combined with NaCl water with the same conductivity that the one on site. In general, there is a need to study the dynamic behaviour of metallurgical residues at lab scale, and to investigate the impact of the water on the electrical properties of the samples (ERT, IP, SIP).

3.1.3.4 Correlation studies

Linear correlations between the geophysical parameters and the concentration of several chemical elements were studied using Pearson's method for the geophysical measurements carried out in the lab and in the field. However, instead of using linear correlations as database, we identified several clusters of different chemical compositions in the cross-plots of chargeability and resistivity. Therefore, we use both geophysical parameters and elemental composition as a training dataset.

Obstacles

The clusters or groups of different composition were identified from 18 samples, which may not be enough data to represent the heterogeneity of the slag heaps. In addition, before computing pairwise correlations between the geophysical and chemical variables we performed a standardization of the data. This allows to compare data of different physical properties as well as the same type of parameters of very different value ranges. Therefore, stronger correlations were observed between geophysical parameters and the concentration of certain elements (< 1 % wt.) compared to those obtained with elements of greater concentrations – which is a fact to be further analyzed.

Opportunities and recommendations

Pairwise correlations proved useful to identify groups of different compositions even only considering the chemical data. They are useful to get an insight of the accuracy of the inverted models (field data) as the correlations between the field and chemical data can be compared with correlations computed between lab geophysical and chemical data. Finally, at this step we can identify which geophysical parameters can discriminate certain chemical elements or materials.

3.1.3.5 Final interpretation: RAPIDM

The interpretation of the ERT/ IP 3D inverted models is done in terms of the groups of different chemical compositions distinguished in the previous step. Then, we use a probabilistic approach using the following data: resistivity and chargeability (inverted) values at the position at which the samples were taken, and the position {*x*, *y*, *z*} of the same samples. Then we compute the joint conditional probabilities of each group in the whole acquisition domain and we perform a classification, i.e., we assign the largest probabilities to the corresponding group in the whole slag heap.

Obstacles

The main obstacle is that there is no ground truth data at depths larger than approximately 6 m. Furthermore, including a spatial trend on the probabilistic approach assumes that similar types of materials were deposited in specific areas of the heap or at specific depths. However, this assumption may not hold true in practice, as the deposition patterns can vary.

Opportunities and recommendations

The probabilistic approach was suitable to perform a classification of the geophysical data acquired in the whole slag heap. As the classification is based on the computation of conditional probabilities the approach also allows to quantify and integrate associated uncertainties in the interpretation model or RAPIDM.

3.2 Additional sites

3.2.1 STPI (BE)

3.2.1.1 Geophysical survey in the field

The geophysical investigation carried out in the field prioritized accessible areas as most of the site surface presents dense vegetation. After the analysis of the historical archives and available information, as well as a couple of site visits we used the following methods: electrical resistivity tomography (ERT), induced polarization (IP) and electromagnetic induction (EMI) mapping.

Obstacles

Several trees, dense vegetation and abrupt discontinuities in the terrain limited the geophysical survey to three ERT/IP profiles and a disrupted EMI coverage. The signal of the DGPS was not continuous and therefore a precise georeferentiation of the ERT/IP profiles was challenging.

Opportunities and recommendations

The EMI mapping allowed to identify lateral contrasts of apparent electrical conductivity and apparent magnetic susceptibility which is useful to delineate zones of interest. We used the conductivity- meter CMD Mini-Explorer that targets an effective depth range of 0.5 m, 1 m and 1.8 m. Additionally, as the objective is to estimate volumes of potentially valuable materials, we used the ERT/IP profiling methods and the EMI mapping method to have vertical and lateral coverage respectively. Note that if we want to compare the electrical conductivities of EMI and

ERT, the EMI data needs to be inverted. In this case, we recommend to select an adequate setup (e.g., electrode spacing, profile length) that eases the comparison of both methods (or integration if for instance we use the inverted ERT model to calibrate the EMI data).

3.2.1.2 Targeted sampling

The targeted sampling was designed from the inverted ERT/IP models and the EMI mapping, where several vertical and lateral contrasts were observed. 16 pits were excavated across the site with depths up to around 4- 5 m and four boreholes were drilled (or monitored if a previous piezometer was present).

Obstacles

The position of several pits had to be shifted due to limited excavator accessibility.

Opportunities and recommendations

Manual sampling may be an option in the sites with reduced accessibility. Hand auger soil sampling could provide subsurface information at shallow depths.

3.2.1.3 Laboratory measurements

The excavations made on the site and the observations reported in the trench/piezometer logs revealed only little amount of slag within the backfill deposits. Therefore, only three samples were selected for laboratory geophysical measurements.

Obstacles

No obstacles were found when measuring ERT and IP in the laboratory.

Opportunities and recommendations

As the first visual assessment during the sampling and chemical analysis indicated that there was a limited quantity of slags, we only measured ERT and (time-domain) IP in the laboratory. The use of these two methods may be enough to associate the largest values of chargeability with the materials with the largest Fe content.

3.2.1.4 Correlation studies

In this site it was not possible to study the correlations between the geophysical measurements in the laboratory and the chemical analysis. Instead, we performed a classification of the field data using categories previously defined from the pits logs.

Obstacles

There was a limited number of samples that were measured in the laboratory and therefore it was not possible to establish correlations with the chemical analysis.

Opportunities and recommendations

We recommend to analyse the results of the targeted sampling before conducting subsequent chemical analysis and geophysical measurements in the laboratory. A first visual inspection in the sampling survey (or borehole logs) may be enough to assess the potential quantity of materials of interest. If the site presents a low quantity of metallurgical residues, correlation studies for laboratory measurements may not be needed but instead the geophysical field data can be interpreted in terms of the materials observed in the excavations.

3.2.1.5 Final interpretation: RAPIDM

First, we summarized and selected the main materials or categories described in the logs of pits and piezometers. Four categories were considered: topsoil, backfill, clayey loam and loam with gravel (hereafter referred as gravel). First, we used the electrical resistivity and chargeability parameters co-located with the excavated trial pits to train a model and classify the remaining geophysical parameters in the whole domain via a machine learning approach. We then used the results provided from the previous classification and the description of trial pits/piezometers to train a new network to identify the presence or absence of backfill based on the EMI data (both apparent conductivity and susceptibility). The results of the EMI classification were used to impose a zero thickness on the backfill layer where no backfill was present. With this constraint and using the results of the ERT/IP classification as well as the ground truth data, the volume of backfill could be estimated by interpolation.

Obstacles

The volume estimation uses the apparent electrical conductivity and apparent magnetic susceptibility of the EMI mapping. Therefore, the depths are a rough estimation as data inversion is needed to have electrical conductivities at punctual depths.

Opportunities and recommendations

We find suitable the approach of combining ate least one profiling method and one mapping method to estimate volume(s) of materials of interest. When using EMI mapping, data calibration (possibly using ERT) and data inversion can lead to better defined vertical boundaries compared to those obtained using only the apparent electrical conductivities. Lastly, we recommend to carefully select the mapping device or setup of the profiling method to have depths of exploration (and spatial resolution) accordingly to the context of the site.

3.2.2 Nyrstar Auby (FR)

3.2.2.1 Geophysical survey in the field

The site is composed of two different waste disposals: 1) active ponds situated North of a canal, where slags from hydrometallurgy (mainly goethite) are still deposed, 2) closed ponds South of the canal, where slags from hydrometallurgy (goethite and jarosite) but also from former pyrometallurgy treatment were deposited. Geophysical surveys were conducted on both sites: magnetic field and surficial magnetic susceptibility measurements, Electrical Resistivity and Induced Polarization tomographies, as well as characterization with Electromagnetic Induction (EMI) tools.

Obstacles

On the North ponds, the main obstacle was the industrial activity where caterpillars spread the waste in ponds. This co-activity with high risk of accident has limited the area for the geophysical survey (only a short test was conducted).

On the South ponds, an insulating tar membrane covers the whole ponds, prohibiting the use of electrical methods. We took advantage of a period of refurbishment of the ponds cover, to be authorized to pierce the tar cover.

Opportunities and recommendations

The magnetic properties of hydrometallurgy slags are quite homogeneous at the pond scale. The magnetic survey doesn't show any specific layering. Nevertheless, magnetic properties of wastes from the pyrometallurgy period were not characterized.

At contrary, electrical properties of wastes from hydrometallurgy and pyrometallurgy are very contrasted. Electrical chargeability and resistivity are different in each surveyed pond, potentially highlighting modifications in the industrial process of zinc ores.

The EMI survey shows the same variation in electrical resistivity as the ERT survey, and is more convenient in environment with insulating cover, such as a tar membrane. However, it is recommended to use ERT and especially Induced Polarization Tomography (IPT) because the vertical resolution is much higher than with EMI and thin layers are visible on ERT and IPT results. Moreover, the electrical chargeability is a parameter characterizing the metallic compounds and shows specific layering in waste deposits.

3.2.2.2 Targeted sampling

Based on the geophysical survey carried out on the former ponds, 7 boreholes were drilled on wastes with specific geophysical signatures. Cores were extracted from the field, and the holes were refiled with bentonite to ensure the stability of the wastes.

Obstacles

As for the geophysical survey, we took advantage of the ponds cover refurbishment to pierce the insulating tar membrane. In normal times, no authorization could be guaranteed for drilling.

Despite careful management of wastes during the activity of the ponds, some unknown layers with hard material were discovered during drilling in the central pond that prevented from reaching deeper wastes with specific geophysical signatures. The core drilling machine was not adapted to cross a hard level. No chemical characterization (including metallic content) and no textural characterization will be possible on these wastes.

Opportunities and recommendations

The choice of the drilling machine is fundamental in sampling strategy. Cores extracted are recommended, but the core drilling tools must be adapted to the materials, as we are working in wastes where unexpected materials could be discovered.

The historical description must be as detailed as possible to avoid any unforeseen events.

3.2.2.3 Laboratory measurements

Following the drilling campaign, samples were only available in March 2023. Laboratory measurements were not run yet. They are planned for July 2023.

3.2.2.4 Final interpretation

Obstacles

At this stage of the study, no comparison between the geophysical results and the core samples laboratory analysis have been made. This point is fundamental to interpret the geophysical signatures, and to calculate the volume of the different wastes identified by the geophysical survey.

Opportunities and recommendations

It seems difficult to characterize such slags from geomagnetic properties, which look quite homogeneous with no structures.

ERT and IPT are the main geophysical techniques recommended for the characterization of slags derived from hydrometallurgical and pyrometallurgical treatments. The two parameters (electrical resistivity and electrical chargeability), as well as the Metal Factor calculated from these parameters, show internal structures in the waste ponds, that are characteristic of chemical composition variations.

EMI is convenient when an insulting membrane prevent the use of electrical methods, for which the membrane needs to be pierced. However, the vertical resolution is poor compared to ERT and the chargeability is not measured with the existing probes.

3.2.3 La Campine (BE)

3.2.3.1 Geophysical survey in the field

The site is composed of two closed landfills which are mainly composed of waste slags from the former lead and antimony production. Both landfills are completely covered with a plastic geomembrane and present a cover layer of soil above the upper geomembrane. This limited the geophysical survey to electromagnetic induction method (EMI).

Obstacles

The main obstacle of this site was the presence of a plastic geomembrane that could not be removed nor damaged (as for an ERT acquisition). During the field acquisition, the furrows in the ground of the landfills complicated the EMI acquisition as it is recommended that the antenna of the device remains steady and parallel to the surface. Finally, the EMI data could not be calibrated for example using ERT (Cavalcante Fraga et al. 2019) or ground truth data (Delefortrie et al. 2019) before the inversion.

Opportunities and recommendations

The EMI method is a completely non-invasive geophysical method that proved to be suitable to study both landfills. In the inverted models it was possible to identify lateral and vertical contrast of electrical conductivity, however, it could have been of utility to use the method of GPR to better delineate the cover layer.

3.2.3.2 Targeted sampling

No targeted sampling was carried out in this site due to the planned interim use (solar panels will be installed in the surface of both landfills). Nevertheless, we used the data from a sampling survey and chemical analyses that were conducted in 2017 within Mivamil project. We used the XRF results from samples collected at depths between 1 to 5 m in nine different boreholes.

Obstacles

The short-term planning for the interim use of both landfills did not allow to conduct a targeted sampling and we could not have recent ground data to calibrate and validate the geophysical data.

Opportunities and recommendations

We recommend to design the targeted sampling based on the results of the geophysical acquisition and collect the samples in a date relatively close to the geophysical survey dates and/or in the same season. Nevertheless, this case shows how the methodology can be adapted with the existing elements.

3.2.3.3 Laboratory measurements

Since we did not gather any sample, we were unable to carry out geophysical laboratory measurements. Instead, we relied on pre-existing sampling data and chemical results from 2017.

The results of the XRF analysis were particularly useful as they were available for the majority of the samples. These results indicated a relatively low average concentration of lead and antimony, while demonstrating a higher iron content.

Obstacles

We could not measure geophysical data in the laboratory.

Opportunities and recommendations

The chemical analysis performed in 2017 by KU Leuven showed a complete characterization of the materials in both landfills. In addition to the XRF analysis, scanning electron microscopy (SEM) and elemental analysis were applied for a more detailed metallurgical characterization in terms of oxides and sulphides.

3.2.3.4 Correlation studies

We studied the linear correlations between the inverted electrical conductivity and the concentration of several chemical elements using Pearson’s method.

Obstacles

We did not observe strong positive nor negative correlations between the inverted values of electrical conductivity and the average content of chemical elements such as Fe, Pb and Sb. This could be related to the errors in the EMI data acquisition, processing (lack of data calibration) and inversion, as well as variations in the water content and in-situ oxidative- weathering conditions (compared to those from sampling dates).

Opportunities and recommendations

Pairwise correlations proved useful to identify groups of different composition only considering the chemical data in both landfills. We recommend to study the linear correlation between the different chemical elements using previously standardized data.

3.2.3.5 Final interpretation: RAPIDM

The interpretation of the models of electrical conductivity was done in terms of two groups of different chemical composition. In this case we also applied the probabilistic approach using the following data: electrical conductivity values at the position at which the samples were taken, and the position {*x*, *y*, *z*} of the same samples. Then we compute the joint conditional probabilities of each group in the whole acquisition domain and we perform a classification, i.e., we assign the largest probabilities to the corresponding group in the whole slag heap.

Obstacles

It was not possible to discriminate the concentration of certain chemical elements using only the inverted electrical conductivity. Therefore, including the trend of spatial data or the position of the samples assumes that the materials from a group may be distributed in certain zones of the landfills or at certain depths – which may not be the case.

Opportunities and recommendations

The methodology adapted here to derive the RAPIDM was still useful to study correlations between the chemical elements and to estimate volumes of different types of materials. Nevertheless, we recommend deriving the RAPIDM using ground truth data obtained at dates closer to the geophysical survey and optimizing sample locations based on the geophysical results.

3.2.4 Vieille-Montagne Grâce-Hollogne (BE)

3.2.4.1 Geophysical survey in the field

The site's historical activities were primarily centered around zinc production. Following a site visit and analysis of available data, it was determined that the geophysical investigation should primarily focus on the central area, which was once a slag heap. A large quantity of materials composing the latter has already been used as backfill for highways, resulting in a relatively flat terrain that could be effectively examined using geophysical techniques. The survey was carried out in three distinct stages. Initially, the electromagnetic induction method was employed to map the area of interest. Subsequently, based on the analysis of the obtained maps, several ERT and IP profiles were conducted with the objective of delineating the extent of anthropogenic deposits and identifying zones enriched with metals. In the final stage, a drone equipped with various sensors, including high-resolution and multispectral cameras as well as magnetometers, was deployed to further capture detailed data over the areas investigated.

Obstacles

The presence of abundant vegetation in the zone of interest posed several challenges during the geophysical investigation. Firstly, it hindered the effectiveness of EMI mapping, as the dense vegetation prevented achieving a sufficiently dense grid coverage of the area. Additionally, the vegetation presented obstacles for the multispectral and photogrammetric acquisitions conducted with the drone. The movement of the vegetation caused by wind disrupted the ability to create an accurate 3D model of the site using photogrammetry techniques. Moreover, the vegetation itself acted as a barrier, concealing the underlying soil and reducing the utility of the multispectral data results. Furthermore, the close proximity of a civil airport added complexity to the drone fly-over operations, imposing restrictions or limitations on the survey. The presence of an airport nearby necessitated careful planning and coordination to ensure compliance with regulations and mitigate any potential risks associated with the drone operations.

Furthermore, considering the electrical properties measured and the limited thickness of the slag deposits, GPR could have been a useful addition to determine the vertical extent of the deposits.

Opportunities and recommendations

Despite the challenges faced in obtaining photogrammetric and multispectral data, the magnetometer data collected by the drone proved to be valuable. The magnetometers successfully identified areas exhibiting magnetic anomalies, which, interestingly, coincided with chargeability anomalies, indicating the presence of ferromagnetic materials. Once again, ERT and IP methods emerged as the most effective techniques for discerning and discriminating between different materials.

3.2.4.2 Targeted sampling

Most of the samples were collected at the soil surface at the location where electrical data were acquired, to enable correlation analysis.

Obstacles

The hardness of the soil made it extremely difficult to penetrate and extract samples, limiting the depth at which reliable samples could be obtained. Moreover, the low number of samples collected (8 in total, and only 6 in the area of interest) further restricted the potential for conducting extensive correlation analysis.

Opportunities and recommendations

When planning soil sample collection, it is crucial to take into account the soil hardness to ensure the appropriate selection of techniques for soil collection.

3.2.4.3 Laboratory measurements

The samples were sent for XRF analysis, which enabled the retrieval of the major elements' composition. However, it is important to note that no geophysical measurements of the samples were conducted in the laboratory.

Obstacles

No obstacles were reported for the XRF analysis of the samples.

Opportunities and recommendations

Samples collected offered an opportunity to study the composition of slag associated with Zinc production.

3.2.4.4 Correlation studies

Despite the small number of samples, linear correlations between the geophysical parameters measured in the field and the concentration of several chemical elements were studied using Pearson's method.

Obstacles

The low number of samples collected restricted the potential for conducting extensive correlation analysis. With a limited sample size, the ability to establish robust relationships and correlations between different parameters or variables becomes compromised. Due to the absence of measured geophysical properties of the samples, correlations were studied using field-measured properties, which may introduce a scale effect.

Opportunities and recommendations

A larger sample set would have allowed for more comprehensive statistical analysis, enabling a deeper understanding of the site's characteristics and facilitating more accurate interpretations. However, the use of a simple classification approach provides meaningful information of the slag heap. This non-supervised classification divided the heap in three component : (i) chargeable materials consistent with slag deposits; (ii) resistive layer composed by anthropogenic deposits; and (iii) conductive substratum of loam natural formation (Dumont et al. 2023).

3.2.4.5 Final interpretation: RAPIDM

The geophysical surveys allows to characterize the whole site. Despiste extensive sampling approaches, a first RAPIDM model have been built. The geophysical interpretations rely on boreholes previously made by SPAQuE and the few geochemical analysis. These information allows to provide a first interpretation of the geophysical properties clustering. This first step needs to be improved in order to provide quantitative information of the slag heap composition.

3.2.5 Reppel, Bocholt (BE)

The site investigated in Bocholt was part of a former Arsenic plant. The area investigated is composed of different landfills where residues of arsenic production were dumped. The site has been covered with a geomembrane to prevent transport of contaminants outside the landfills area.

3.2.5.1 Geophysical survey in the field

Geophysical survey was conducted in three steps. First, a drone equipped with two types of sensors (a high-resolution camera and magnetometers) flew over the area of interest. The idea was to build a 3D model of the surface of the site using photogrammetry and detect zones containing potentially a higher content of ferro-magnetic materials. In a second step, a mapping of the site was carried out with EMI. Finally, three ERT-IP profiles were deployed in the landfill to search for potential leaks in the watertight containment system.

Obstacles

The presence of a nearby military airport posed significant challenges to the drone flight, imposing strict limitations on its altitude. Furthermore, the existence of a geomembrane beneath a protective cover layer significantly restricted the effectiveness of electrical methods in providing comprehensive information. It also appears to affect the quality of data.

Opportunities and recommendations

The magnetic map obtained with the drone did not reveal any significant anomalies suggesting the absence of ferromagnetic materials in the landfill. On the other hand, EMI maps exhibited areas of higher conductivity that appeared to be associated with potential leaks in the geomembrane, as also suggested by the results obtained from ERT.

3.2.5.2 Targeted sampling

No targeted sampling could be carried out due to the presence of the geomembrane.

3.2.5.3 Final interpretation

The geophysical survey provides reliable information on area with water leakage through the geomembrane. The water flows through the past arsenic wastes could transport heavy metals to the aquifer and pollute the groundwater. In this case, the geophysics provide information on area where the geomembrane should be inspected and repair in order to avoid any pollution risk for the surrounding population.

4 Conclusions

In this report, we have presented an integrated methodology for geophysical investigations in past metallurgical sites and deposits (PMSD) and the development of a raw materials and pollution distribution model (RAPIDM). The methodology outlined in this report provides a comprehensive framework for conducting effective investigations and characterizing PMSDs. These guidelines aim to assist future researchers, environmental consultants, and policymakers in applying the methodology to other PMSD sites.

The first step in implementing this methodology is to conduct a thorough site assessment. Factors such as site type, materials present, topography, infrastructure, and budget should be carefully considered. This assessment will help in selecting appropriate geophysical methods, sampling strategies, and laboratory analyses that are tailored to the specific site conditions.

The geophysical survey should be planned and executed based on the site assessment, considering investigation objectives, subsurface conditions, and target materials. Using a combination of geophysical techniques is recommended for a comprehensive understanding. Start with a drone survey using different sensors for a rapid site assessment and identification of areas for further investigation. Next, conduct surface mapping methods like EMI in the identified areas to gather information on potential anomalies. For deeper insights, deploy electrical profiling methods to understand subsurface characteristics and detect buried features. Conduct the survey systematically, ensuring adequate coverage while considering practical constraints like vegetation, infrastructure, and access.

Following the geophysical survey, targeted sampling should be carried out to validate and complement the geophysical data. The sampling strategy should be designed based on the results of the geophysical survey to ensure that samples are collected from depths and locations that are most representative of the site. It is important to consider stability of slopes, safety concerns, and budget limitations when planning the sampling campaign.

Laboratory measurements play a crucial role in characterizing the physical and chemical properties of the collected samples. Geophysical measurements such as electrical resistivity, induced polarization, and spectral induced polarization should be performed to determine the subsurface electrical properties. Chemical analyses, including X-ray fluorescence, X-ray diffraction, and inductively coupled plasma spectroscopy, should be conducted to identify the elemental composition, mineralogy, and particle size distribution of the samples.

Correlation studies should be undertaken to establish relationships between geophysical parameters and chemical elements. Statistical analyses, such as linear correlations and clustering techniques, can help identify groups of samples with similar chemical compositions. These correlations provide insights into the spatial distribution of raw materials and pollution within the site.

The interpretation of geophysical data and chemical composition forms the basis for developing RAPIDMs.

In conclusion, the guidelines outlined in this report provide a roadmap for applying the integrated methodology to future geophysical investigations in PMSDs. These guidelines, coupled with site-specific adaptations and considerations, will aid in the efficient characterization of PMSDs and the development of RAPIDMs.

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