

D. I2.3.2. REPORT ON THE POTENTIAL OF EXTRACTING RAW MATERIAL ON SITE

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3 INTRODUCTION

Pompey is one of the three pilot sites of the NWE-REGENERATIS project. It is a former tailing pond owned by the EPFGE (Etablissement Public Foncier de Grand Est, Public Real-Estate Company of Grand Est region). The site has been chosen for two main reasons: (1) it hosted various activities for iron based alloys production; (2) it was just rehabilitated on surface, and historic documentation and investigations are done with respect of the French legislation and threshold values. One of the interest of this site is that it allows testing the REGENERATIS methodologies developed within WPT1 and WPT2 on a site that has already been remediated.

Site works provided access to material to perform lab trials and also allowed on-site geophysical measurements. Two phases of sampling were handled on site: (1) traditional sampling prior to the geophysical investigations; (2) targeted sampling after the geophysical investigations.

A site-specific report on sampling investigation (see DI2.2.2) provided specific data on pre- and post-sampling investigations campaigns that were performed on site. The traditional sampling investigations aims at characterizing the nature, physical and chemical compositions of the wastes at a punctual location. Given the results, the potential of extracting raw material on site was evaluated.

This report discusses the potential of extracting 5 m³ of materials on site, combining different mineral processing technologies. We first present the results of the sampling campaign on site, and then highlight results from a previous study on the potential for extracting zinc (Zn), manganese (Mn) and lead (Pb) from the first layers of wastes in the Pompey tailing pond.

4 PRESENTATION OF THE POMPEY SITE

The Pompey site 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 active from 1870 to 1986. It is 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. The dike delimiting the site was planted with a curtain of black locust trees in 1997. The rest of the pond gradually got covered with diversified deciduous vegetation, more or less dense depending on the area.

The geological substratum of the former tailing pond consists of the Lias marl formations (at 181 m NGF), which are covered by alluvium from the two rivers, composed of coarse siliceous materials (sands, gravel and pebbles) at the base over 3 to 6 m surmounted by finer materials (sands, silts and clays) on 1 to 3 m. These alluvial formations were locally exploited and backfilled with waste rock and iron and steel by-products.

The depth of the deposits in the basin is estimated at around 10 m. The surface of the former pond is estimated to 26 000 m², for a total estimated volume of wastes equal to 260 000 m³.

The waters of the alluvial table would circulate from the channeled Moselle towards the Meurthe, whose level is lower. The piezometric levels measured in 2002 are 187.5 m NGF upstream (South-West of the island) and 184 m NGF downstream (East of the island), the basin surface being at 197 m NGF (ANTEA, 2002).

On site work included a two-stages geophysical campaign. The geophysical measurements allowed to map the variations of geophysical properties laterally and horizontally, over the entire surface of the deposit. These methods provide very valuable and almost continuous information laterally and vertically. However, they are only indirectly linked with the targeted properties, such as concentration

of metallic elements, size the metallic particles... In addition, a two-stages sampling campaign was also conducted: (1) traditional sampling prior to the geophysical investigations within an already-existing pit (fosse in Figure 1), from 0 to 2m deep; (2) targeted sampling based on the geophysical results within 5 selected boreholes, from 0 to 9m deep (see Figure 1). Characterization of the samples was led in terms of the nature, physical and chemical composition of the wastes. The main results are presented in the following section (section 5) of this report.



Figure 1: Map of the different sampling locations on the Pompey site.

5 RESULTS OF THE SAMPLING CAMPAIGNS

5.1 RESULTS OF THE 1ST SAMPLING CAMPAIGN: 0-2M

We first ran a sampling campaign prior to the geophysical investigations. A pit, excavated in 2010, was already available on site (see “fosse” in Figure 1). The first samples of soils to analyze were thus taken within the pit, in the first two meters. Two types of samples were extracted:

- 17 soil samples were taken every 20 cm from 0.2 m to 1.8 m depth. Two different sides of the pit were investigated (noted P2 and P4) with portable X-ray fluorescence spectroscopy analysis at BRGM: 9 samples for P2 and 8 samples for P4.
- 2 soil samples were taken in the pit at 2 depths: [1.0 - 1.1] m (S1 sample) and [1.65 - 1.80] m (S2 sample) for deeper laboratory analysis in CTP’s laboratory. The choice of these two depths were dictated by preliminary soil analysis from Huot (2013) that detected higher content of Pb and Mn at 1 m; and of Zn at 1.8 m.

In the following paragraphs, we synthesize the main conclusions already established in the report DI2.2.2. For further details, please refer to the report DI2.2.2.

Mineral processing results

Based on the mineral processing analysis, differences can be highlighted between the samples S1 and S2, in terms of mineral processing analysis:

- At 1.1 m depth, the techno-soil is composed mainly of **fine particles** (below 600 μm), and has a much higher portion of magnetic particles, revealing the potential presence of **ferrous residues**
- At 1.7 m depth, the techno-soil is composed mainly of **coarse particles** (greater than 10 mm), and within its finer portions, has a lower proportion of magnetic particles, indicating a potential **lower proportion of ferrous residues**.

XRF/pXRF analysis

Based on the pXRF and XRF results, three different layers can be observed within the studied pit (see Figure 2):

- From 0 to 0.8 m: the highest metallic element concentration is iron (Fe) with values ranging from 20 to 30 %
- From 0.8 to 1.4 m: The Fe concentration drops, while the concentration of the other metallic elements increase (especially Pb – around 4%, and Cu – around 0.04%)
- From 1.4 to 1.8 m: The Mn concentration is the highest (7-20 %), while Fe ranges from 2 to 10 %

The Fe and Mn concentrations are not varying the same way with depth, indicating that they are not present in their alloy forms, which was expected taking into account the history of the former plant: production of ferromanganese.

Regarding the chemical analysis on each separate particle size and magnetic fraction, several observations can be highlighted:

- except for iron, which concentrated in particle size fractions smaller than 600 microns, the sieving of the material do not seem to have any impact on its chemical composition.
- the magnetic fraction is mainly composed of iron (28-30%) with various concentrations of other non-ferrous metals. Manganese was more concentrated in the low-magnetic and non-magnetic fractions even if its grade (between 1 and 12% w/w) remained low compared to that observed in an ore (~40%). This observation confirmed that manganese was not in alloy with iron.

Although the analyzed samples contain various metals, their grades are too low to justify any economical interest in recovering them within the first 2 m of soil, in the particular location of the trench pit.

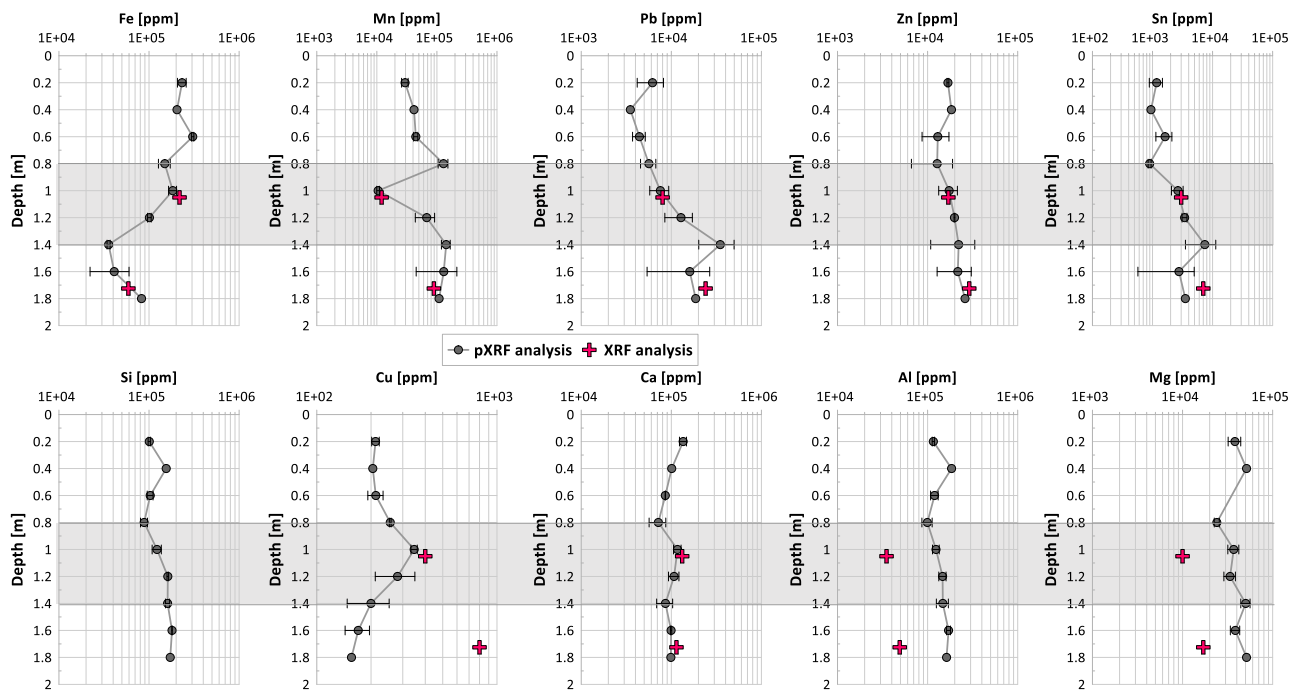


Figure 2: Variation of concentration of different selected chemical elements versus depth for measured using the pXRF (NITON) method, and the XRF method. The pXRF results displayed are the median and standard deviation estimated between two measurements made on two different samples extracted at the same depth, within the same pit, on 2 different profiles (P2 and P4 – see DI2.2.2). The grey area between 0.8 and 1.4 m reflects the pXRF interpretation in terms of horizontal layers with different chemical compositions.

Conclusions

Based on mineral processing and chemical composition analysis, three different layers can be observed from 0 to 2 m, within the trench pit:

- From 0 to 0.8 m: the highest metallic element concentration is Fe with values ranging from 20 to 30 %.
- From 0.8 to 1.4 m: The Fe concentration is dropping, while the concentration of the other metallic elements are increasing (especially Pb – around 4%, and Cu – around 0.04%). In the center of this layer, the soil is mainly composed of fine particles and has a much higher portion of magnetic particles within which iron is the major element, revealing the potential presence of ferrous residues.
- From 1.4 to 1.8 m: The Mn concentration is the highest (7-20 %), while Fe is ranging from 2 to 10 %. In this layer, the soil is mainly composed of coarse particles (greater than 10 mm), and within its finer portions, has a lower proportion of magnetic particles, indicating a lower proportion of ferrous residues.

The Fe and Mn concentrations are not varying the same way with depth, indicating that they are not present in their alloy forms, which was expected taking into account the history of the former plant.

Although the analyzed samples contain various metals, their grades are too low to justify any economic interest in recovering them within the first 2 m of soil, in the particular location of the trench pit. Post-sampling investigations are scheduled to:

- explore the recovering potential of metals deeper in the deposit
- analyze the recovering potential of the deposit in terms of eco-catalysis.

5.2 RESULTS OF THE 2ND SAMPLING CAMPAIGN: 0-9M

No heavy machinery is allowed to enter the site. We must therefore consider the option of a lightweight auger. IXSANE proposes to study the idea of carrying out a few sampling points with an auger that can cross up to 5 m of land depending on the soil conditions.

Following the first geophysical results, it appears that the potentially most interesting soil layer starts at 2-3 m deep and up to 8-9 m deep (see deliverable DI2.2.1). It would therefore be interesting to take samples from this layer in the second sampling campaign, both on the NNW and SSE sides of the former tailing pond. Several sampling locations have been decided based on these geophysical observations

4 samples were taken by IXANE at two different locations I1 and I2 (see Figure 1): IXSANE extracted the samples using a lightweight auger that can cross up to 5 m of land depending on the soil conditions. The samples were taken at two different depth intervals: [0-0.5] m and [3-4] m

45 samples were taken by BRGM using a light core drill for depths ranging between 0 and 9.2 m at 4 different locations (see Figure 1): 11 samples for FP1 from 0 to 9.2 m, 10 samples for FP2 from 0 to 8.2 m, 12 samples for FP3 from 0 to 9.2 m, and 12 samples for FP4 from 0 to 9.2 m

pXRF analysis

The chemical characterization led both by IXANE and the BRGM on the selected samples are very consistent with the results of the pre-sampling investigations (see Figure 3 and deliverable DI2.2.2). The analysis can thus be considered as reliable and used for further interpretations.

Three main layers can be distinguished for all the metallic chemical elements discussed here:

- **From the altitude 195 m to 194 m (thickness ~1m):** The heavy metal concentrations are mostly lower than in the layer below, but not as low as within the bottom layer. This first layer of techno-soil is the one the most subject to anthropic shuffle (uncontrolled dumping...). As the site is covered by vegetation, it is also remodeled by roots and other life forms that can potentially mobilize some of the metallic elements.

An exception can be made for sounding FP1, where the concentration of Mn, Zn, Pb and Cu is higher at the surface, and then drops at 194.5m. This observation can be linked to the presence of anthropic wastes posterior to the closure of the settling pond that form a mound where metal bars and concrete block have been observed.

- **From the altitude 194 m to a variable altitude ranging from 188.6 to 186.7 m (5.4 m < thickness < 7.3 m):** The concentration of each of the metallic elements is the highest. This layer is interpreted as the main deposits from the former tailing pond.

It can be noted that the thickness of the deposits is higher in the South of the field site (FP1 and FP4) than in the north (FP2 and FP3).

It can also be noted that this main deposit layer can be divided into 2: Around 191.5 m, a drop in the concentrations of the different metallic elements can be observed. This can be indicative of a change in the nature of deposit, linked with the history of the plant (change in composition of the metals processed...)

- **Below a variable altitude ranging from 188.6 to 186.7 m:** the concentration of all the metallic elements drops drastically from several order of magnitude. This trend might be indicative of the bottom of the deposit. The lower concentration in heavy metals and higher concentration in silicon might indicate the transition with a layer of natural alluvial deposits.

The drop is observed at a depth ranging between 6.4 and 8.3 m depending on the location of the sounding. According to this set of data, the thickness of the deposit is thus lower than what was expected before the analysis (estimated at 10 m depth).

The additional agronomical analysis indicate that both the top layer (0-0.5 m depth) and the middle of the deposit layer (3-4 m depth) have good fertility rates. The water conductivity being higher in deeper layer, the fertility is a little better for the top soil. On the contrary, the concentrations of metallic elements in Cu, Hg, Pb and Zn is higher for the deeper samples. Both analyzed depth are thus interesting for eco-catalyst productions.

5.3 RECOMMENDATIONS

Pre- and post-sampling investigations were carried out on samples taken at different depths from a former siderurgical settling basin, potentially concentrated in metals (Fe, Zn, Mn, Pb, etc.).

The chemical analysis carried by various laboratories (CTP, IXANE and BRGM) are indicating consistent metallic element concentrations, indicating the reliability of the data obtained.

Several layers can be observed looking at the concentration of the different metallic elements, with a drop between 188.6 and 186.7 m of altitude depending on the sampling locations. This drop could be indicative of the transition between the anthropic deposits and the natural alluvial deposits. It is observed at depth ranging between 6.4 and 8.3 m, which is lower than the expected depth of the deposits, previously estimated to around 10 m. The total recoverable material is thus lower than expected.

Furthermore, although these samples contained various metals, their grades are still too low to justify any economic interest in recovering them.

On the contrary, the fertility rates measured are good on the tested samples. This type of deposits seems thus to be more suitable for recovery by eco-catalysis.

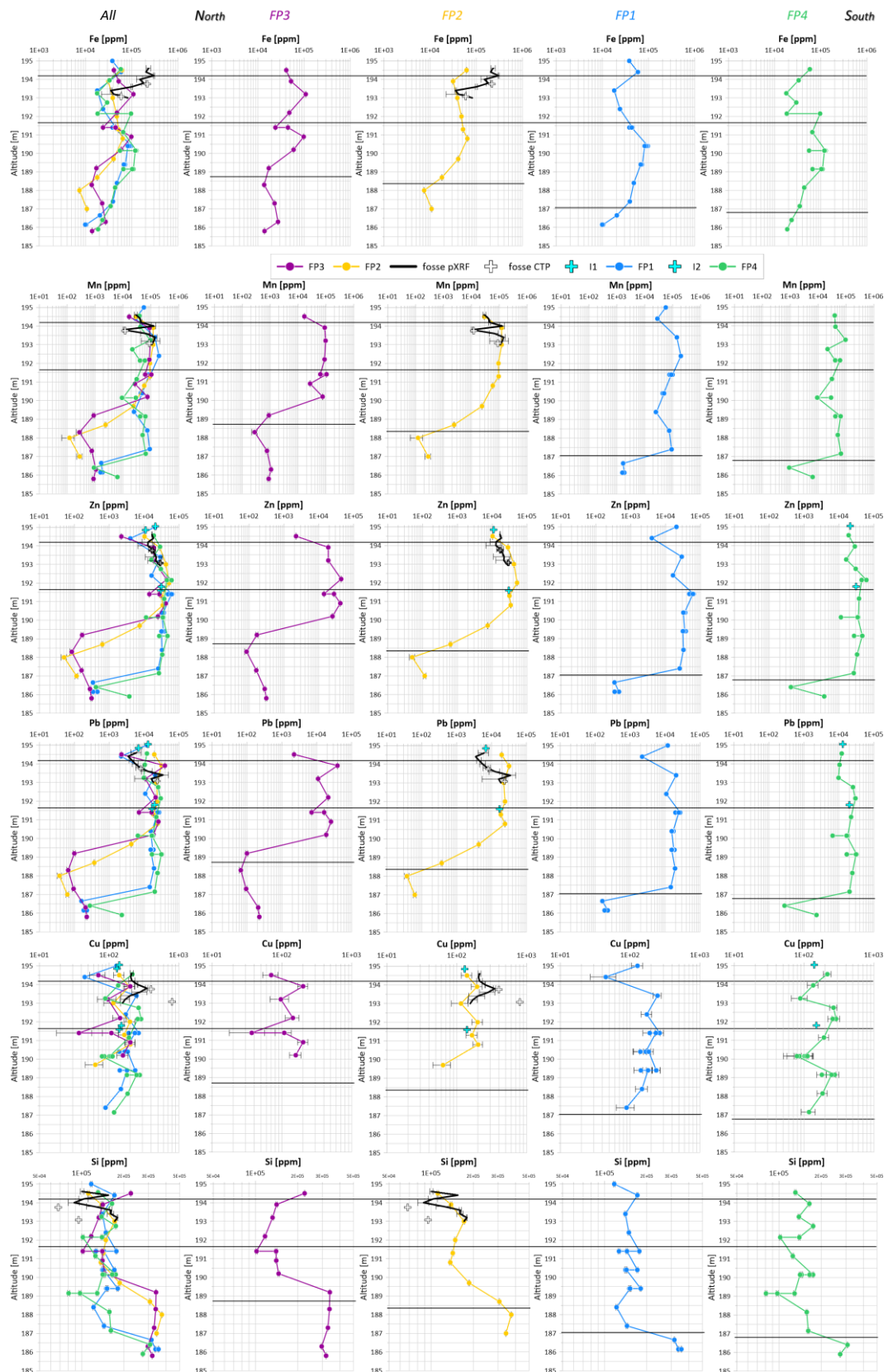


Figure 3 : Variation of concentration of different selected chemical elements (Fe, Mn, Zn, Pb, Cu, Si) versus altitude for all soundings (FP1 to FP4, I1 and I2) from the north to the South of the site (see Figure 1). These results are compared to pre-sampling analysis within the pit: (1) pXRF analysis on two profiles down to 1.8 m depth; and (2) XRF results on two samples S1 and S2 analyzed at CTP (fosse CTP).

6 PREVIOUS STUDY ON THE POTENTIAL OF EXTRACTING RAW MATERIAL ON SITE

A PhD thesis was defended in 2015 by Julien Mocellin on :”Secondary metal sources, recovery of zinc, manganese and lead from pyrometallurgical sludge by hydrometallurgical processing”. In his PhD, he studied the development of a full and efficient hydrometallurgical process to recover zinc, manganese and lead from samples extracted in Pompey. Pyrometallurgical methods have been used to exploit Zn, Mn and Pb-ores since the mid-19th century. However, these methods are expensive, and energy-intensive. Hydrometallurgy processes were thus chosen instead of pyrometallurgical processes to reduce the costs of the processes, as well as their ecological footprint (see Deliverable DT1.3.4).

In Mocellin (2015), four samples were taken at the pit location (see fosse in Figure 1) at 4 different depths (see [Figure 4](#), samples A, B, C and D). A 5th sample was created at the lab scale by mixing the 4 different sampling location together. The general characteristics of these deposits are that: (1) they are rich in metallic elements, especially zinc (Zn), manganese (Mn) and lead (Pb); (2) their matrix is very complex (see [Table 1](#)).

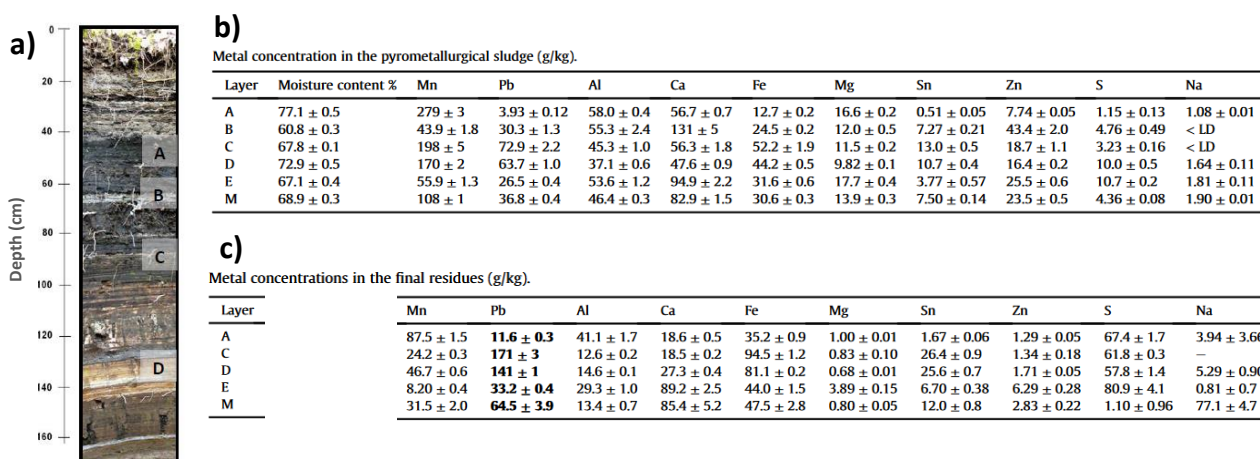


Figure 4: a) location of the samples within the technosoil profile (from Huot, 2013 and Huot et al., 2014b); Table 1: b) Metal concentration in the selected samples from the Pompey site (A, B, C, D, E), and the artificially mixed sample (M) (from Mocellin, 2015) Table 2: c) Metal concentration in the final residues, after the hydrometallurgical process was implemented (from Mocellin, 2015)

The hydrometallurgical processes chosen by Mocellin (2015) include several steps described in Figure 5: (1) pretreatment; (2) Selective Leaching; (3) Leachate purification; (4) Metal recovery; (5) Mineral refinement.

Steps (2), (3) and (4) have been very thoroughly studied in Mocellin (2015), Mocellin et al. (2015) and Mocellin et al. (2017). We will summarize the main outputs in the next parts of this report.

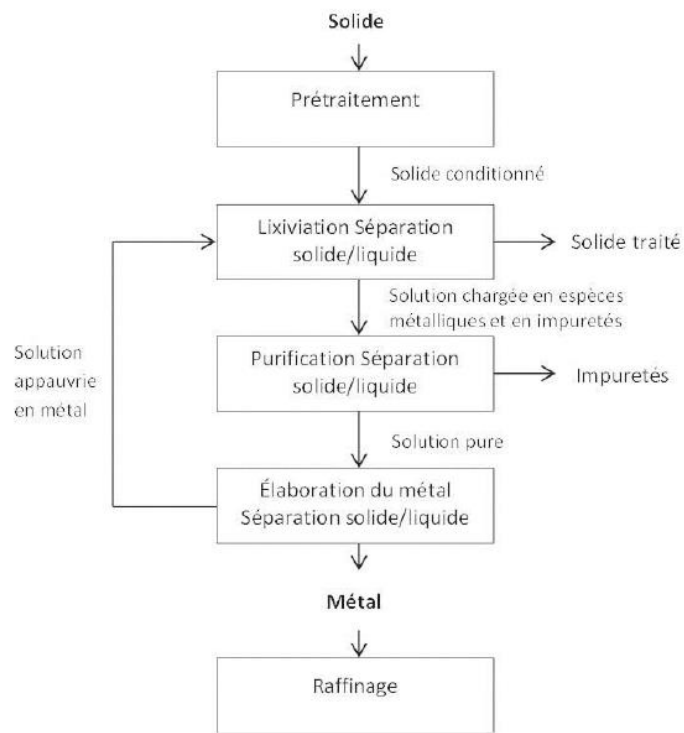


Figure 5 : General hydrometallurgical process scheme (from Rizet et al., 2000)

6.1 SELECTIVE LEACHING

The selective leaching part of the hydrometallurgical process was studied in details in Mocellin et al. (2015).

The selective leaching process was broken down into two distinct parts:

6.1.1 Zn extraction

The study of the yield of Zn extraction was made using four factors and four levels of an experimental design called Box-Behnken design. In statistic, this type of design is built for response surface methodology that explores the relationships between: (1) exploratory variables (in this case the experimental conditions of the leaching such as concentration of sulfuric acid or temperature), and (2) response variable (in this case the yield of Zn extraction). This type of designs allows to explore all the experimental conditions possible, and not just the ones experimentally tested, in order to optimize the experimental conditions to reach a specific yield of extraction.

For Zn extraction, the optimum conditions chosen are (see Figure 6 a)):

- Concentration of sulfuric acid: 0.25 mol/L
- Pulp density (or solid/liquid ratio): 10%
- Extraction temperature: 20°C
- 3 stages of leaching of 20 minutes each

6.1.2 Mn extraction

The study of the yield of Mn extraction was made using the same type of Box-Behnken design.

For Mn extraction, the optimum conditions chosen are (see Figure 6Figure 7 b)):

- Concentration of sulfuric acid: 0.25 mol/L
- $\text{Na}_2\text{S}_2\text{O}_5/\text{Mn}$ stoichiometry: 1
- Leaching time: 120 min
- 2 stages of leaching

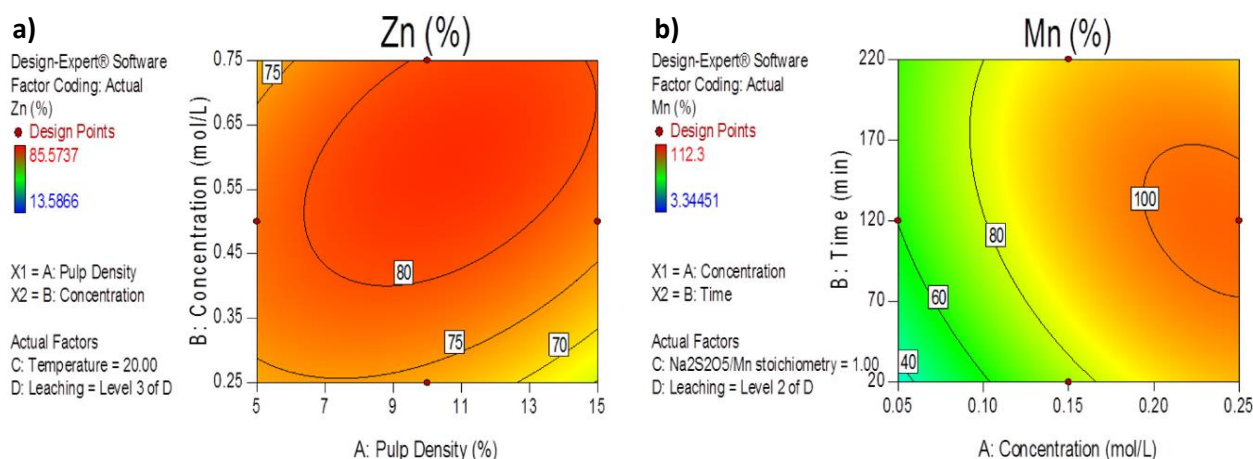


Figure 6: Example of Box-Behnken response surface design chosen to optimize: a) Zn extraction and b) Mn extraction (from Mocellin et al, 2015)

This optimization study allows to hope for a potential yield of Zn extraction equal to 75%, and a potential yield of Mn extraction equal to 100%

After further experiments described in Mocellin et al. (2017), the experimental recovery rate of Zn after the leaching step ranges between 81 and 100%, and the one of Mn between 77 and 96%. For the Mn selective leaching phase, the authors particularly recommend to:

- Fix pH range from 3.5 and 4.2
- Get redox potentials lower than 400 mV
- Use $\text{Na}_2\text{S}_2\text{O}_5$ and H_2SO_4 as reducing agents

6.2 LEACHATE PURIFICATION

The detailed description of the process involved in the removal of aluminum (Al) and iron (Fe) from the Zn leach liquor is described Figure 7. The purification is made using a chemical precipitation process at a pH of 4.8 with NaOH reagent.

The detailed description of the process involved in the removal of aluminum (Al), zinc (Zn), and lead (Pb) from the Mn leach liquor is described Figure 7. The purification is made using a chemical precipitation process at a pH of 5.6.

6.3 METAL RECOVERY

The **zinc precipitation** is run first (see Figure 7). Using twice the stoichiometry value of Na_2S , Zn precipitates in its sphalerite form (FeS).

The **manganese precipitation** is run next (see Figure 7). Using Na_2CO_3 as the precipitation agent and a basic pH equal to 8.5, Mn precipitates in its rhodochrosite form (MnCO_3).

At the end of the PhD work of Mocellin (2015), the Pb recovery in the residues remaining after the Zn and Mn precipitation is not yet addressed.

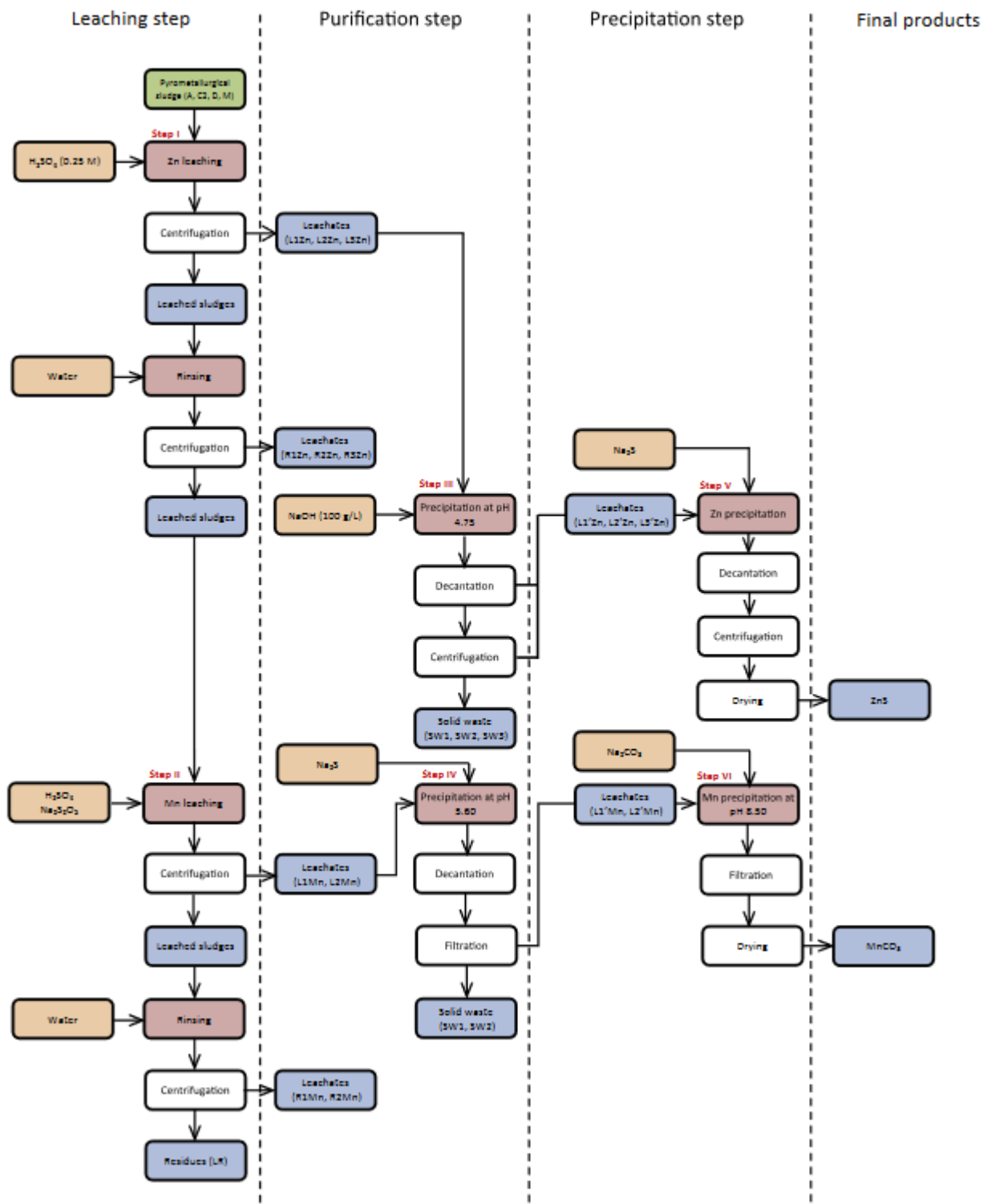


Figure 7: Flow sheet of the hydrometallurgical process tested (from Mocellin et al., 2017)

6.4 GLOBAL PROCESS AND METHODOLOGY

The methodology followed during the work of Mocellin (2015) is shown in Figure 8. The studies allowed to initially optimize the leaching, purification and development stages of the process at a laboratory scale on a specific sludge sample in order to optimize the operation conditions. Tests at the laboratory scale have been coupled with tests at a pilote test scale, in order to assess process applicability and robustness.

The investigations of the leachate purification and metal recovery were tested at lab scale first, for only one layer of the technosoil profile (profile C- see [Figure 4](#)). The test of all the layers on different technosoil profiles were then run both at lab scale, and pilot scale.

The results obtained made it possible to carry out an economical study.

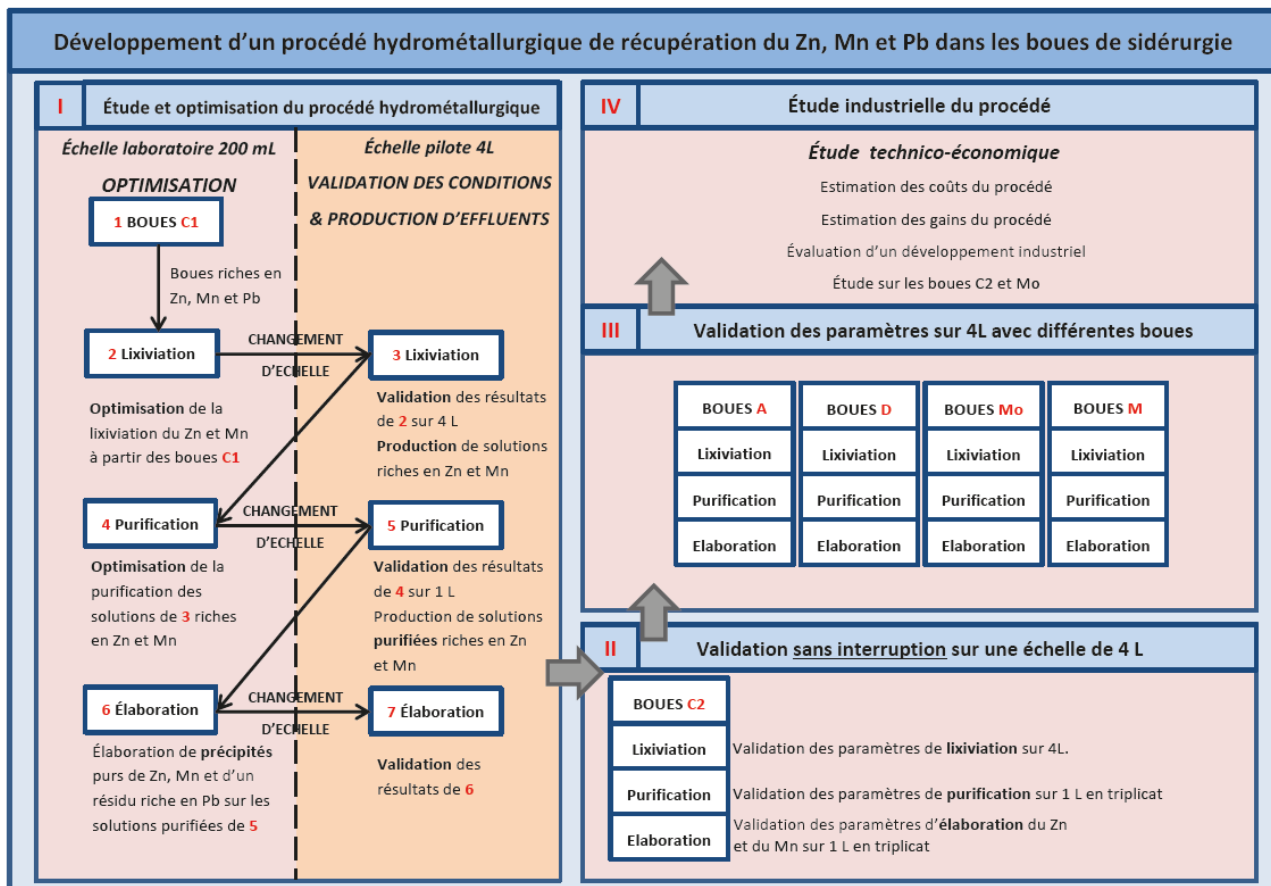


Figure 8: Diagram of the methodological path followed during the development of the process (from Mocellin, 2015)

6.5 ECONOMICAL CONSIDERATIONS

After a careful study of the prices of the raw metallic elements revalorized, in 2015, and the total costs for the full hydrometallurgical process involved, the following observations were made by Mocellin et al. (2017) (see Table 3):

- The income generated by the production of Zn and Mn is equal to 365.82 \$Can/ton
- The expenses spend to realize the hydrometalurcal process studied are equal to 552.74 \$Can/ton
- There is thus a **deficit of 186.92 \$Can/ton**

Compared to remediation techniques or containment measures, this cost is equivalent to the cost incurred by landfilling (187 \$Can/ton), and above the costs of on-site impoundments, such as adding a geo-membrane cover to the tailing pond (between 30 and 163 \$Can/ton). For cases where the settling pond wastes need to be transformed into landfill, this process might start to be interesting.

If the prices of the metals continue to increase over time, the ratio expenses spent versus income generated might evolve.

Mocellin et al (2017) also cite several ideas to reduce even more the costs of the metal recovery:

- For the Mn extraction: replace the $\text{Na}_2\text{S}_2\text{O}_5$ with SO_2 produced through burning of sulfure
- To save steps in the Mn extraction, recover Mn in the rinsing solutions L1 and L2.

These solutions might help decrease the gap between the expenses and the income. However, in the current status, the extraction process is not equilibrated, and even less profitable.

Table 3 : Total cost and revenue per ton of the residue for layer D (from Mocellin et al., 2017)

Direct operational cost	Residue C2 (\$Can/ton)
Chemical products	
Sulfuric acid (H_2SO_4)	–53.89 \$
Sodium carbonate(Na_2CO_3)	–51.11 \$
Sodium métabisulfite ($Na_2S_2O_5$)	–137.76 \$
Sodium sulfide	–34.34 \$
Manpower	
Operation and supervision	43.00 \$
Utility	
Electricity	–65.02 \$
Water	–22.56 \$
Divers	–31.68 \$
Subtotal	–458.91 \$
Indirect and overhead costs	–93.83 \$
Total costs of operations	–552.74 \$
Revenues	
Zinc	20.61 \$
Manganese	345.21 \$
Total operating revenues	365.82 \$
Balance sheet	–186.92 \$

7 CONCLUSIONS

In this report we synthesis the results of the sampling campaigns run during the REGENERATIS project on Pompey site and draw conclusions about the non-profitability of the metal extraction on site to this day. A first analysis on the chemical element concentration versus depths from 0 to 9 m was led. It shows high metal content (especially for Fe, Mn, Zn and Pb), but according to CTP, not high enough to start trying to valorize them using extraction techniques.

We also report on the results of a PhD work led by J. Mocellin (see Mocellin, 2015; Mocellin et al., 2015 and Mocellin et al., 2017) that developed a specific hydrometallurgical process to extract zinc, manganese and lead from the techno- soil layers (0 - 2 m) on the Pompey site. The complexity of the matrix of the wastes present in the settling pond deposits is challenging. However, they show a detailed process that allows to extract more than 75% of Zn and Mn, following 3 optimized steps: (1) selective leaching ; (2) leachate purification; (3) metal recovery. An economical analysis on the benefits of the planned rehabilitation is also presented. The study conclude that, at the present time, the hydrometallurgical process developed is not yet profitable on the Pompey site. However, with

the prices of metals increasing and the growing importance of state critical metal production, the developed processes might be suitable and useful in the future.

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Atelier Mémoire Ouvrière : website of a group of enthusiasts who bring te story of the Carreau minier from Val de Fer at Neuves-Maisons to life, <http://amo.fjep.pagesperso-orange.fr/>

BASOL: database on polluted or potentially polluted sites and soils calling for action by public authorities, for preventive or curative purposes, <http://basol.developpement-durable.gouv.fr/>

Community of communes of the Pompey basin website: <http://www.bassinpompey.fr/>

Géoportail: website of the National Institute of Geographic and Forest Information (IGN), <http://www.geoportail.gouv.fr>

Infoterre : website gathering all available BRGM data: geological maps from 1: 1,000,000 to 1: 50,000, files from the Basement Data Bank and geological logs, maps of natural and industrial risks, data on groundwater, etc., <https://infoterre.brgm.fr/>