

# ASSESSING METAL RECOVERY OPPORTUNITIES THROUGH BIOLEACHING FROM PAST METALLURGICAL SITES AND WASTE DEPOSITS: UK CASE STUDY

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## ABSTRACT

Recovery of metals from industrial and commercial areas (also called brownfields), closed landfill sites, and marginal land areas are critical for future sustainable development and reducing environmental risks posed by historic contaminated sites. This project focuses on the opportunities for metal resources recovery from past metallurgical sites and deposits (PMSD). The Teesside site located in the UK is assessed for its potential for resource recovery of raw and secondary raw materials via bioleaching. Assessment of the physicochemical parameters (conductivity, pH, moisture and ash content), with metal content being carried out using ICP-MS analysis along with initial testing using a portable X-ray fluorescence spectrometer as a rapid measurement tool. Fe (469,700 mg/kg), Ca (25,900 mg/kg) and Zn (14,600 mg/kg) were the most dominant elements present in the samples with relatively high concentrations of Mn (8,600 mg/kg), Si (3,000 mg/kg) and Pb (2,400 mg/kg) observed. The pXRF results demonstrated minimal variance (<10%) from the ICP-MS results. A preliminary assessment of bioleaching technology using *Acidithiobacillus ferrooxidans* was carried out. Bioleaching was performed at 5% pulp density with 22 g/L energy source and 10% (v/v) inoculum at pH 1.5. After bioleaching, 100% of Ti and Cu, 32% of Zn and 24% of Mn was removed from the sample material, highlighting opportunities for the recovery of such metals through bioleaching processes.

## 1. INTRODUCTION

Past metallurgical sites and deposits [PMSD] comprise around 13% of the 2.5 million contaminated sites across Europe (Panagos et al. 2013). The contaminated soil and the deposits represent not only a long-term environmental risk, but also an opportunity to recover valuable metals through innovative processes. An ongoing Interreg project, REGENERATIS on of Past Metallurgical Sites and Deposits through innovative circularity for raw materials [REGENERATIS], aims to enable viable recovery of such metals. As part of this project, a pilot site (one of three for the project) located in the UK is being investigated to demonstrate rapid/non-destructive measurement techniques to assess recovery potential and bio/chemical leaching processes for metal recovery.

This work is part of a programme of research aimed at recovering metals from contaminated sites and closed landfills, thus is part of the enhanced landfill mining ap-

proach reported in previous studies. Metals are a valuable resource and are critical to sustainable development in clean energy technologies. Assessment of opportunities and risks are an important first step to understanding the challenges a site poses and determining the correct sequence of managing the site for resource recovery. Work being carried out within the REGENERATIS project will harmonise existing databases from across North West Europe and develop an open-access platform to enable new business opportunities for metal recovery from PMSDs.

Metallurgical slag, sludge and dust are the by-products of the metal industry and the main reason for the contamination in PMSD. These by-products originate from the iron and steel industry (ferrous/steel making by-products) and base metal industry (non-ferrous). In this study, due to the PMSD being a former iron and steel industrial site, samples coming from the deposits are expected to be a ferrous slag, sludge and flue dust. There has been more interest in base



metal by-products due to its high content of non-ferrous metals (Binnemans et al., 2020). Recently, the recycling of steelmaking by-products started to gain more attention due to extended environmental regulations (Wang et al., 2019a). In 2020, the global steel production reached 1,828 million tonnes, and it is estimated that approximately 10-15% of steel slag is generated per unit of crude steel produced (OECD, 2021; Piatak et al., 2015). Slags are widely used as construction material (Binnemans et al., 2020). In Europe, approximately 17% of steel slag goes into stockpiles (Hocheng, Su and Jadhav, 2014). Sludge and dust are harder to valorise than slag, owing to their high zinc content (Binnemans et al., 2020). Therefore, stockpiling is used as the only option by the iron and steel industry. However, slag, sludge, and dust deposits pose an environmental risk due to metal release and occupy valuable land (Wang et al., 2019b), yet they can offer metal recovery opportunities.

Biological leaching methods can be used to recover metal from metallurgical slags. Bioleaching technology emerges as a promising biohydrometallurgical approach due to its low energy and raw material requirement, low operating cost, simplicity and environmental friendliness (Asghari et al., 2013; Mishra et al., 2008). In favourable conditions, specific microorganisms, some bacteria and fungi, can solubilise metals from metal bearing materials through their bio-oxidation mechanisms, this process is called bioleaching. Several bioleaching studies have been published for non-ferrous slag do date, yet there are a few bioleaching studies for ferrous slags. The main reason is that the ferrous slag contains small amounts of base metal compared to non-ferrous slag. Hocheng et al., (2014) used pure *Acidithiobacillus thiooxidans* (*At. thiooxidans*), *Acidithiobacillus ferrooxidans* (*At. ferrooxidans*), and *Aspergillus niger* (*A. niger*) cultures to dissolve metals from electric arc furnace (EAF) steel slag sample. They reported that the repeated bioleaching cycles increased metal recovery from 28% to 75%, from 14% to 60% and from 11% to 27%, for Mg, Zn and Cu, respectively, by *At. Thiooxidans*. Gomes et al. (2018) successfully recovered 100% of Al, 84% of Cr and 8% of V from basic oxygen furnace (BOF) steel slag in 22 days by performing column bioleaching using mixed acidophilic bacteria. Thus, it can be hypothesised that metals can be dissolved from steel slag present at Teesside, by using bioleaching technology.

This study contributes to effort to characterise the historical iron and steel legacy wastes in the UK and to assess resource recovery opportunities (Riley et al., 2020). The paper presents initial findings following the characterisation of samples taken from the Teesside Steelworks, demonstrating the validity of using handheld X-ray fluorescence [XRF] as a rapid measurement for determining potentially recoverable metals at a large PMSD of significant national importance. Further to this, *A. ferrooxidans*, was selected and acclimatised to different sample matrices for bioleaching. Preliminary bioleaching experiment was performed at 5% pulp density with 22 g/L energy source and 10% (v/v) inoculum at pH 1.5. After bioleaching 100% of Ti and Cu, 32% of Zn and 24% of Mn was removed. Fe and Bi did not dissolve. So that iron was secured in the BOF material. The results are encouraging to turn stockpiled BOF sludge and

dust into a secondary iron source alongside recover some metals from it. Further study is needed to understand the leaching kinetic and to optimise the process.

## 2. MATERIALS AND METHODS

### 2.1 Site

The South Tees Development Corporation [STDC] site is a large site (1500 ha) with a 160-year history of iron and steel production and the processing of finished products. Approximately 224 ha of land is associated with ironmaking, and 14 ha is associated with the South Bank Coke Ovens. In addition, 26 ha is associated with the South Lackenby Effluent Management System (SLEMS). It comprises large areas of Redcar, Lackenby, Grangetown and South Bank to the South of the River Tees.

The site has been used, at varying periods, 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. The materials have also co-mingled with natural ground materials. This includes dispersal in soil, rock, clay, silt, and other materials arising from its tidal estuary location. The stratigraphy is, therefore, varied and complex. The Teesside PMSD site area is shown in Figure 1.

A total of fifteen bulk (>20kg each) samples were randomly extracted from a large volume (>100 tonnes) of material stored in a covered facility awaiting export this material originated from the land fill and waste management area in figure 1 and 2 part of the SLEMS area. these samples were a type of ferrous slag known as flue dust or sludge which is a by-product of cleaning the gas emissions of a BOS plant (Binnemans et al., 2020)..

### 2.2 Physio-chemical characterisation

Samples were air-dried, crushed, and sieved using a 2 mm mesh (Figure 2). Sieved samples were air-dried again to be used for characterisation experiments which include the determination of the pH (ISO 10390:2005), electrical conductivity (ISO 11265:1994), dry matter and water content (ISO 11465:1993), loss on ignition (450°C 4 hours) (BS EN 13039:2000) available phosphorus (ISO 11263:1994) and water holding capacity (ISO 11274:1998). Total carbon and total nitrogen in Teesside samples were determined by thermal decomposition at least 900°C in the presence of oxygen gas, by Elementar (British Standard BS EN 13654-2:2001). Mastersizer was used to determine the particle size distribution under 2 mm particle size. All characterisation experiments were done in triplicate. The elemental composition of Teesside sample No. 1 was determined by Marchwood Scientific Services by using inductively coupled plasma-mass spectrometry (ICP-MS) for Al, As, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, K, Mn, Hg, Na, Ni, Sb, Se, Si, Sn, Ti, Tl, V, Zn (Table 1).

### 2.3 Rapid measurement tools

Rapid determination of metal concentrations was determined using a portable X-Ray fluorescence spectrom-



**FIGURE 1:** Landfill and waste management areas, including the SLEMS. Red box highlights area of sample origin.

ter (pXRF) (Olympus Delta Premium USA). Three samples (~200 g) were collected in LDPE bags from each of the 15x bulk samples. These were measured in triplicate through the bags in the integrated test stand accessories using the Geochem mode for 30 seconds in real-time, on both beams 1 and 2. No pre-treatment was applied to the samples.

#### **2.4 A. *ferrooxidans* adaptation study and bioleaching**

*A. ferrooxidans* (DSM 583) was sourced from the Leibniz Institute (DSMZ), Braunschweig (Germany). The reference strain 1% (v/v) was cultivated in 250 ml Erlenmeyer flask containing 99 ml of optimized 4.5 K salt medium (Chen et al., 2015). The optimized 4.5 K salt medium consists of two solutions. Solution A was prepared by adding 2.00 g of  $(\text{NH}_4)_2\text{SO}_4$ , 0.25 g of  $\text{K}_2\text{HPO}_4$ , 0.25 g of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , 0.10 g of KCl and 0.01 g of  $\text{Ca}(\text{NO}_3)_2$  into a 700 ml of deionised water. solution B contains 22.2 g of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (4.44 g  $\text{Fe}^{+2}$ ) in 300 ml deionised water. Both solution were adjusted at pH 2 using 5 M  $\text{H}_2\text{SO}_4$ . After pH adjustment, Solution A was autoclaved at 121°C for 15 minutes. Solution B was instead filtered using 0.2  $\mu\text{m}$  filter. the culture was incubated at 30°C on an orbital shaker at 150 rpm. Once the cul-

ture oxidation-reduction potential (ORP) reaches  $\geq 600$  mV, it was assumed that the culture reaches the log phase, and the cell number is approximately  $1 \times 10^8$  cells/ml. (Chen et al., 2015; Muddana and Baral, 2021). After several incuba-



**FIGURE 2:** Teesside sample No. 1 after sieving through 2 mm mesh.

tion step 10% (v/v) pure culture was inoculated in 250 ml Erlenmeyer flask containing 90 ml of optimized 4.5 K salt medium for adaptation study.

Microbial adaptation is a well-known acclimatisation technique in the bioleaching process to increase the bioleaching potential and enhance metal toxicity tolerance of the selected microorganism (Chen et al., 2015; Muddana and Baral, 2021). To adapt *A. ferrooxidans* to the Teesside materials, the solid/liquid ratio was increased gradually as follows: Once the ORP of the pure culture was  $\geq 600$  mV, 0.5% w/v of material was added to the optimized 4.5 K medium (Figure 3). The culture was then left to recover until the ORP was again  $\geq 600$  mV, which indicates that acclimation was successful. Then 10% (v/v) of the adapted inoculum was collected and re-incubated with a fresh growth medium containing 1% (w/v) of material. Once the ORP was  $\geq 600$  mV, the same operation was repeated for 2.5% and 5% (w/v) addition of materials. The pH and ORP were monitored on a daily basis and pH was adjusted to desired value. For 0.5% w/v pulp density, pH was selected as 1.5, while for the other pulp densities pH was 1.75. Rotation speed was increased from 150 rpm to 160 rpm for 5% w/v pulp density at day 3 and 4 due to the solid particles have been observed to precipitate as their mass increases. All adaptation conditions were carried out in duplicate. Control conditions consisted of the culture medium without microorganism and material incubated at 30°C and 150 rpm.

For bioleaching, after adaptation completed for 5% w/v, BOF sludge and dust, 10% (v/v) of the adapted culture was then cultivated in a 250 ml Erlenmeyer flask containing 90 ml of optimized 4.5 K salt medium at 30°C and 150 rpm at pH 1.75. After two incubation step 10% (v/v) adapted culture was inoculated for the same condition only pH was selected as 1.5. When ORP reached  $\geq 600$  mV, 5 g of BOF sludge and dust was added into the flask. Flask was incubated for 16 days. Everyday, ORP was measured, and pH was adjusted at 1.5 using 5 M  $H_2SO_4$ . Ferrous iron consumption and the oxidation of  $Fe^{+2}$  to  $Fe^{+3}$  is considered as an indication

of microbial growth (Roy et al., 2021).  $Fe^{+2}$  concentration was analysed by titration against  $K_2Cr_2O_7$  on day 1, 3, 5, 8, 11, 16 (Third et al., 2000). End of the experiment the residue was filtrated by Whatman grade 2 filter paper, washed with deionized water, air dried for 24 hours and dried at 60°C for 24 hours. Residue was then analysed by p-XRF.

### 3. RESULTS AND DISCUSSION

#### 3.1 Sample characterisation

Teesside steelworks is a historical iron and steelwork plant. Based on the sample origin, Teesside samples can be categorized as a type of ferrous slag known as flue dust or sludge which is a by-product of cleaning the gas emissions of a BOS plant (Binnemans et al., 2020). The pH of the samples varied between 8.96 and 9.26, and the electrical conductivity ranged between 92.20 and 405.67 S/m (Figure 4). Piatak et al., (2015) stated pH value can vary between 7.40 and 9.13 for ferrous slag, and 4.14 and 7 for non-ferrous slag which is in good agreement with our pH values (Figure 4).

The dry matter and water content (%) results are given in Figure 5. The dry matter and water content analysis was performed at 105°C for 24 hours. Determination of mass loss on ignition (%) was performed at 450°C for 4 hours. Negative values were found for ten samples out of fifteen. Negative results are due to the high iron content in the Teesside samples (Figure 5 and Table 1). Vandenberghe et al., (2010) analysed the mass loss on ignition of an iron-rich coal fly ash and bottom, observing that iron oxidation caused weight gain and it overcame the weight loss of carbon. As such, negative results cannot be excluded for iron-rich ashes.

The value of total nitrogen content ranged from 0.065 – 0.117% which is relatively low when compared to carbon (Figure 6). Total carbon content was relatively high and ranged from 4.26% – 7.47%. Total hydrogen content is also categorised as low ranging from 0.314 – 0.453%. The standard deviation is minimal in total carbon, total ni-

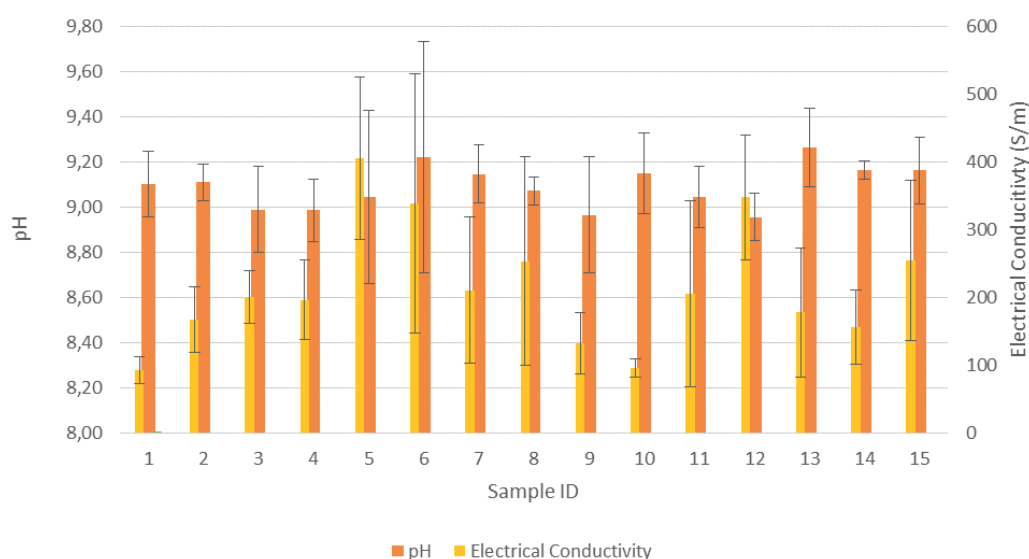


FIGURE 3: Bar charts showing mean pH and electrical conductivity (S/m) of 15 samples collected from Teesside, UK.

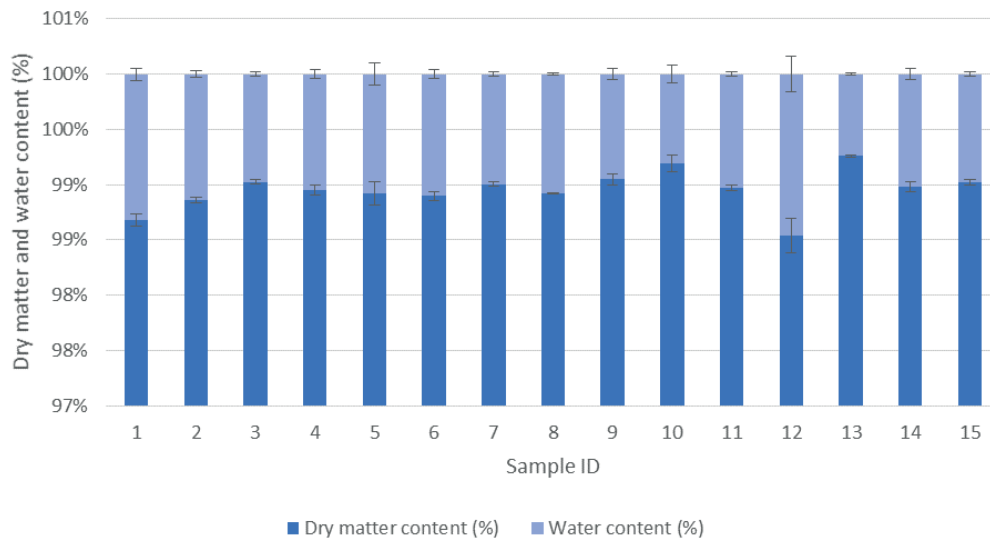


FIGURE 4: Bar charts showing mean dry matter content (%) and water content of 15 samples collected from Teesside, UK.

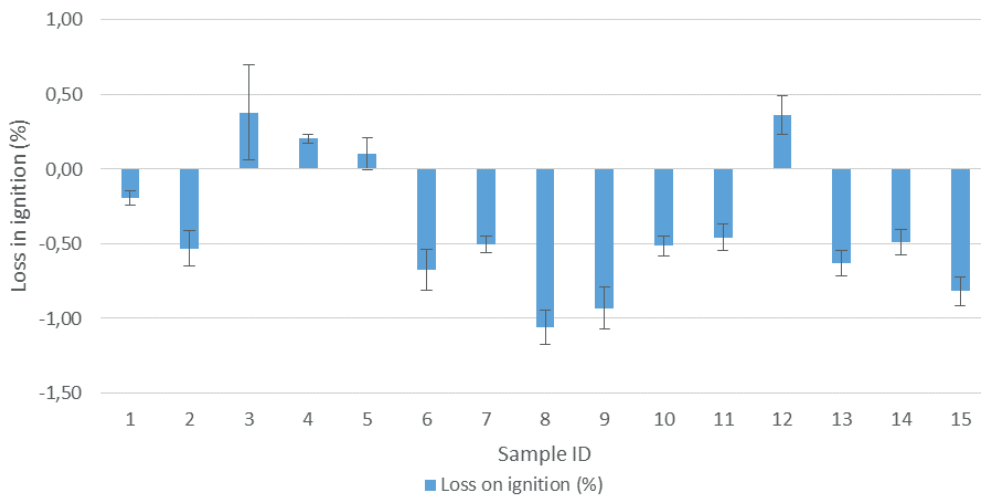


FIGURE 5: Bar charts showing mean loss on ignition (%) of 15 samples collected from Teesside, UK.

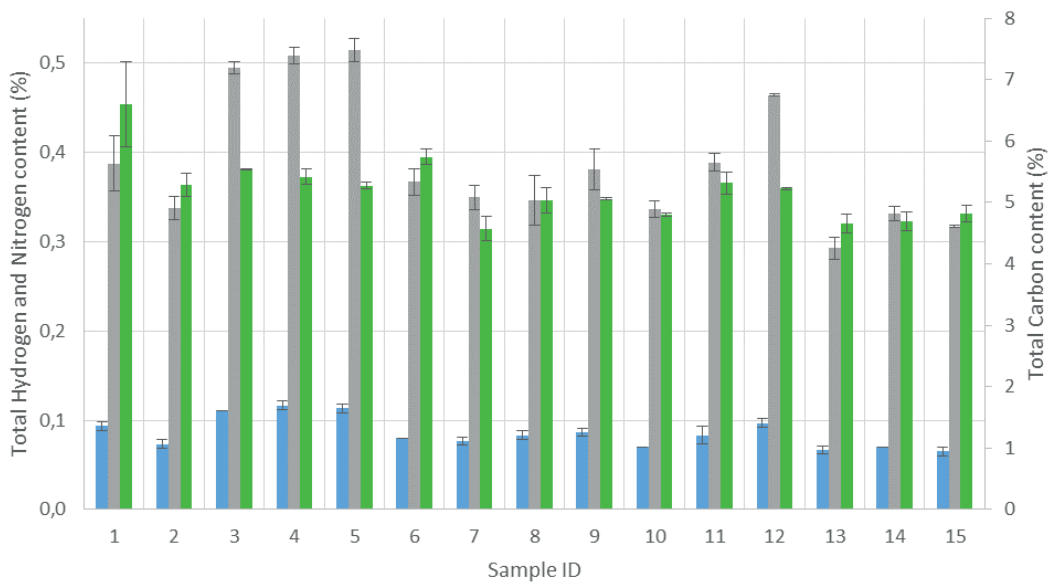


FIGURE 6: Bar charts showing mean total nitrogen, hydrogen, and carbon (%) of 15 samples collected from Teesside, UK.

**TABLE 1:** Comparison of the elemental composition result of Teesside Sample with literature values.

	Fe slag (average, mg/kg)	Pre-1900 Fe Slag (average, mg/kg)	Steel Slag (average, mg/kg)	BOF steel slag (mg/kg)	Primary BOF dust and sludge (average wt%)	Secondary/fine BOF dust and sludge (average wt%)	This study Teesside Sample No.1 (mg/kg, except C which is wt%)
	(Piatak et al., 2015)	(Piatak et al., 2015)	(Piatak et al., 2015)	(Gomes et al., 2018)	(Binnemans et al., 2020)	(Binnemans et al., 2020)	
C	-	-	-	-	0.9	1.9	4
As	6.5	4.5	24.6	10 ± 4	-	-	9.4
Ba	557	806	366	30 ± 10	-	-	99
Cd	-	0.3	14.7	1.6 ± 0.3	-	-	71
Co	9.447	58.6	7.88	-	-	-	8.5
Cr	1,032	9.1	4,798	100 ± 20	-	-	147
Cu	15.9	65.5	114	20 ± 4	-	-	134
Ni	14.4	7.24	153	40 ± 20	-	-	54
Pb	21.7	73.9	126	500 ± 200	0.06	0.1	2424
Zn	79.5	15.4	748	30 ± 4	0.3	1.6	14644
Al	-	-	-	0.4 ± 0.07	-	-	<1
Be	-	-	-	-	-	-	<1
Ca	-	-	-	-	-	-	25873
Fe	-	-	-	-	73.7	60.5	468716
Ga	-	-	-	10 ± 2	-	-	-
Hg	-	-	-	-	-	-	<1
K	-	-	-	200 ± 200	-	-	386
Li	-	-	-	4 ± 1	-	-	-
Mn	-	-	-	1000 ± 20	-	-	8632
Mo	-	-	-	2 ± 1	-	-	-
Na	-	-	-	300 ± 70	-	-	90
Sb	-	-	-	-	-	-	6.2
Se	-	-	-	-	-	-	<1
Si	-	-	-	-	-	-	3041
Sn	-	-	-	-	-	-	61
Sr	-	-	-	30 ± 10	-	-	-
Ti	-	-	-	0.002 ± 0.0003	-	-	138
Tl	-	-	-	-	-	-	7.0
V	-	-	-	40 ± 10	-	-	46

trogen, and total hydrogen (0.191, 0.004, and 0.011 respectively). The volume density of particles falls predominantly within fine sand (0.075 - 0.42 mm) and medium sand (0.42 - 2.0) boundaries.

Available P values ranged between 6.67 and 10.50 mg/kg. The average value of the maximum water holding capacity values of the 15 samples found was 48.9%.

The elemental composition analysis results belong to Teesside sample No.1 (Table 1). Fe (468,716 mg/kg), Ca (25,873 mg/kg) and Zn (14,644 mg/kg) were the most dominant elements. Sample also has high Mn, Pb and Si content. When elemental composition of Teesside samples was compared with average literature values for Fe slag, Pre-1900 Fe slag and Steel slag (Piatak et al. 2015; Gomes et al. 2018), high amount of Zn, Mn, Pb and Cd content were noticed. Besides, Fe, Pb and Zn values are

close to the secondary/fine BOF dust and sludge values (average). Binnemans et al., (2020) stated that average Zn values are 0.2 wt% in BF (Blast furnace) dusts and 1.5 wt% in BF sludges. In addition, Pb values in BF sludges varies between 0.1 and 0.9 wt%. According to this, BF sludge and dust values are also close the Teesside sample values; however, low carbon content associated with BOF dusts and sludges. For BF sludge and dust carbon content varies between 10% and 50%. Therefore, based on the characterisation experiments and literature review, more specifically, Teesside sample can be categorised as BOF sludge and dust (Binnemans et al., 2020).

### 3.2 pXRF results

Using pXRF as a rapid analysis tool, the most prevalent element in samples collected from Teesside was Fe mak-

ing up more than 40% w/w of samples (Figure 7) as expected from ICP analysis (Table 1). The next most prevalent element was Zn ranging from 14,909-9,252 mg/kg, followed by Mn, Bi, Cr, Rb, Cu, Ag, As, Ba, Cd, Co, Hg, Mo, Nb, Ni, Th, U, V, W, Y, Zr were also measured but were mostly below the limit of detection. The remaining mass of approximately 50% w/w were light elements (LE) i.e. elements with an atomic number < 12 (Mg), which are outside the pXRF measurement range and elements between atomic mass 12 (Mg) and 21 (Sc). These elements could not be accurately quantified due to the thickness of the polyethylene in the sample bags which scatters the fluorescence produced by these elements. As a result, the absence of signal measured for elements 12 to 21 would increase the predicted concentration of elements <12. Minimal variation (<10%) in concentrations between samples was observed for most elements, with notable exceptions of Zn and Ti. The high variation of Ti is potentially due to the interference of the polyethylene sample bags, which are reported by the manufacturer to interfere with elements lighter than Sc. Studies have shown the effects of different film thickness and composition have varying effects on lighter elements and calibrations should be carried out on new films before they are employed as equations can be employed to account for the suppression of fluorescence signal (Henke, Gullikson and Davis, 2022) (Ravansari et al., 2020).

This study presents the first known case of measuring BOS flue dust found on a brownfield site with pXRF; proving to be a reliable instrument for elements heavier than Ti (atomic number >22), taking 60 seconds to take each measurement. This can, however, be increased to 120 seconds to further increase accuracy, and the use of disposable Prolene sample cups would allow accurate quantification of elements between atomic mass 12 (Mg) and 21 (Sc).

Sample size is a key limitation of the device as it only measures a small surface area (approx. 2 cm<sup>2</sup>), meaning that if the site under investigation has a high heterogeneity,

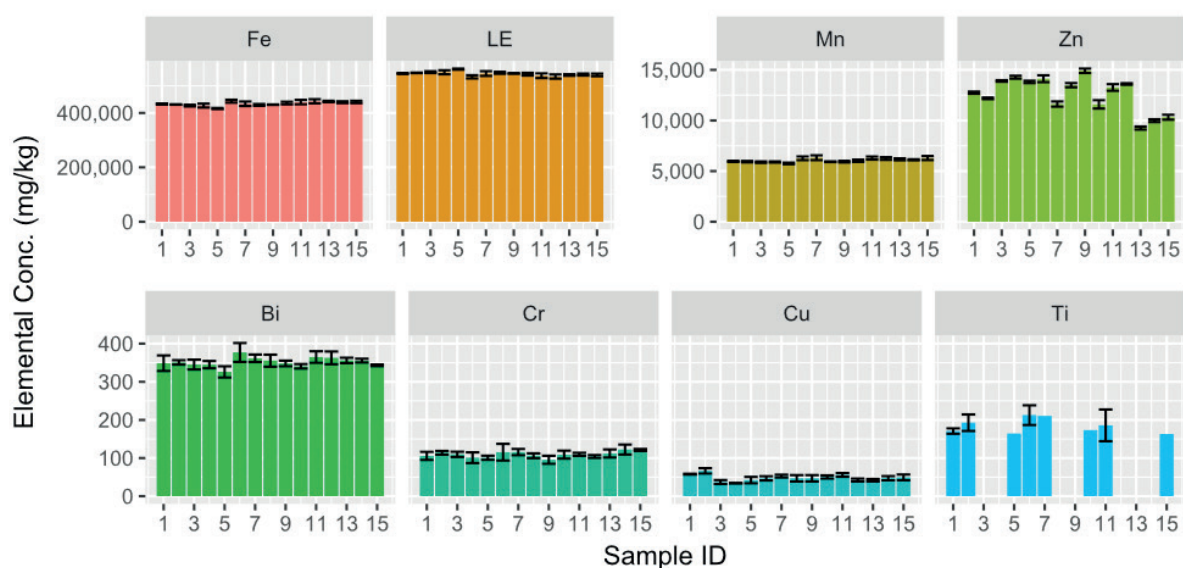
many repeat measurements will be needed to overcome this limitation (Ravansari et al., 2020). In this study, triplicate measurements were performed on the triplicate grab samples from each barrel, and it was found that, on average, measurements within each grab sample (~200g) had a relative standard deviation of 8% hence it was assumed triplicate measurements would provide representative results across this site.

The strong performance of the pXRF in this study is likely due to the fine particle size and low heterogeneity of flue dust, as well as its low organic matter content, which all result from the method of production and are factors that are well reported to interfere with pXRF measurements (Ravansari et al., 2020). The performance of the pXRF would be unlikely to suffer any significant drop in accuracy when applied in-situ to this material, provided that the environmental conditions were not overly wet on the day of measurement as no pre-treatment was applied to the samples. In addition, when applied in-situ, elements 12 (Mg) to 21 (Sc) could be accurately measured due to the lack of a sample container suppressing their signal.

### 3.3 A. *ferrooxidans* adaptation study and bioleaching

An overview of *A. ferrooxidans* adaptation to the BOF sludge and dust is shown in Figure 8. For all pulp densities, after Teesside material addition, a sharp decrease in the ORP was observed (Figure 9). On average for every adaptation cycle, it took 2 days for the culture to recover and reach an ORP  $\geq$  600 mV. Overall, it took 15 days to adapt *A. ferrooxidans* up to 5% (w/v) Teesside material. This adapted culture was used for the batch bioleaching experiment.

The adapted culture was used for bioleaching study. First, adopted culture was cultivated without BOF sludge and dust for 2 days. Over the experiment pH was adjusted at 1.5 by using 5 M H<sub>2</sub>SO<sub>4</sub>, everyday. After two days, the



**FIGURE 7:** Bar charts showing mean elemental concentration (mg/kg) of 15 samples collected from Teesside, UK, error bars show standard deviation over nine measurements, y-axis have three different scales for the varying concentrations of elements.

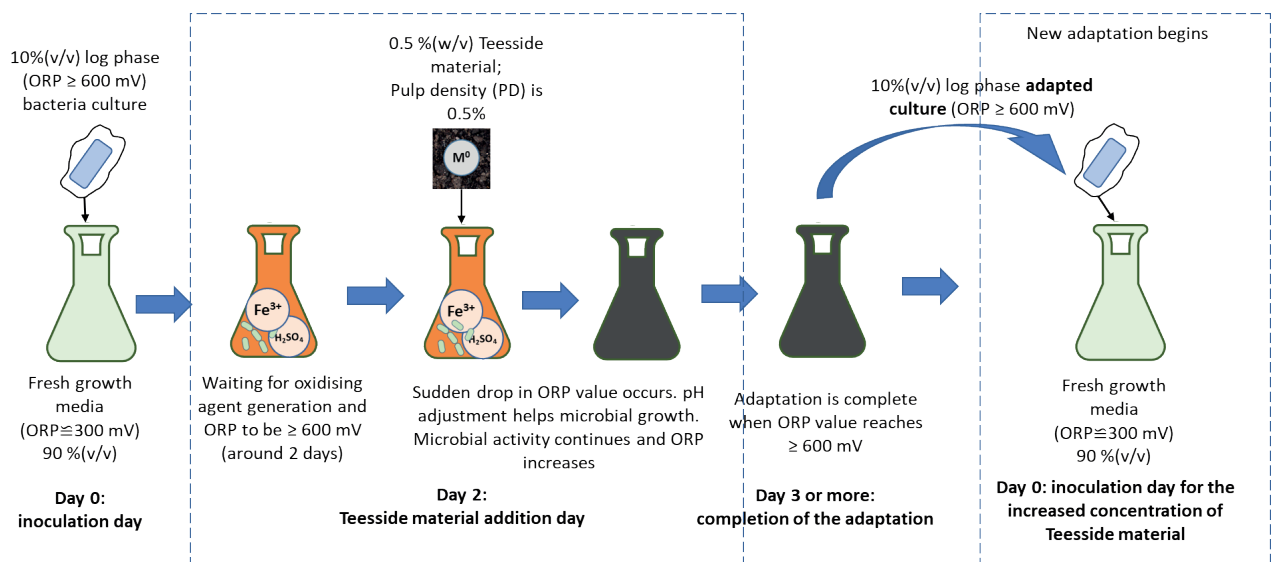
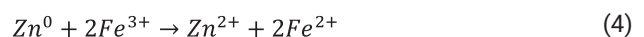
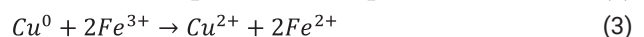
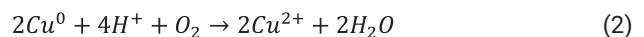
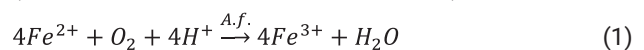


FIGURE 8: The illustration of the bioleaching adaptation study.

ORP value reached 640 mV (Figure 10). Then, 5 grams of BOF material was added to the flask. Before and after material addition pH was adjusted at 1.5. After material addition, sharp decrease was observed on the ORP value, and it dropped 500 mV. *A. ferrooxidans* continued to live and to transform  $Fe^{2+}$  to  $Fe^{3+}$ . Thus, ORP value increased on the following day, day 3, reached 570 mV. On that day more than 95% of the ferrous iron have been consumed by the bacteria. Next day, day 4, ORP hit the 600 mV, then, started to slightly decrease. Changing on ORP value and ferrous iron was compatible with the typical trend (Chen et al., 2015; Muddanna and Baral, 2021).

*A. ferrooxidans* is a chemolithotrophic bacteria which utilises ferrous iron and reduced sulphur compounds to grow and produces ferric iron and sulphuric acid as metabolites (Srichandan et al., 2020). Ferric iron and sulphuric

acid are useful oxidisers which dissolve metals such as copper (Cu) and zinc (Zn) by following reactions (Eq. 1-4) (Chen et al., 2015; Srichandan et al., 2019).



After bioleaching, BOF residue was collected and analysed with p-XRF to have initial insight into remaining metals. Results are shown on the Table 2. According to initial results, 32.1% of Zn and 23.9% of Mn was removed by bioleaching. Dissolution rate can be improved by parameter optimisation (Tavakoli et al. 2017; Zare Tavakoli et al., 2017a, 2017b). The initial concentration of Ti was 138 and

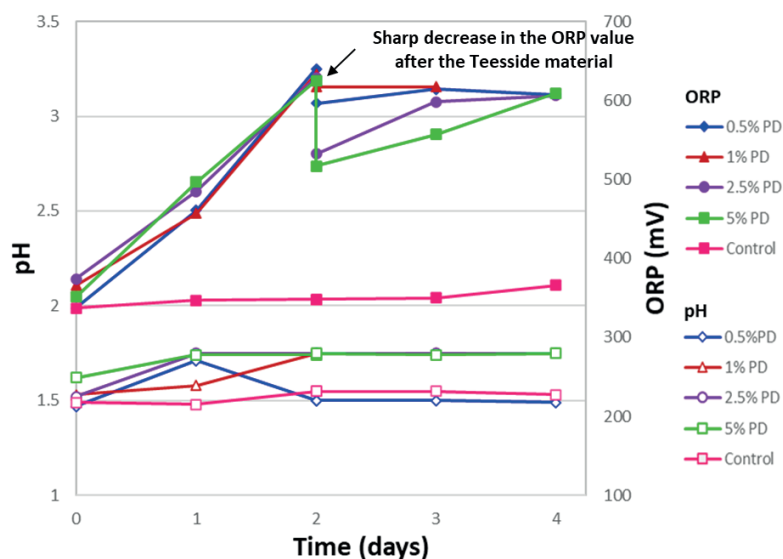
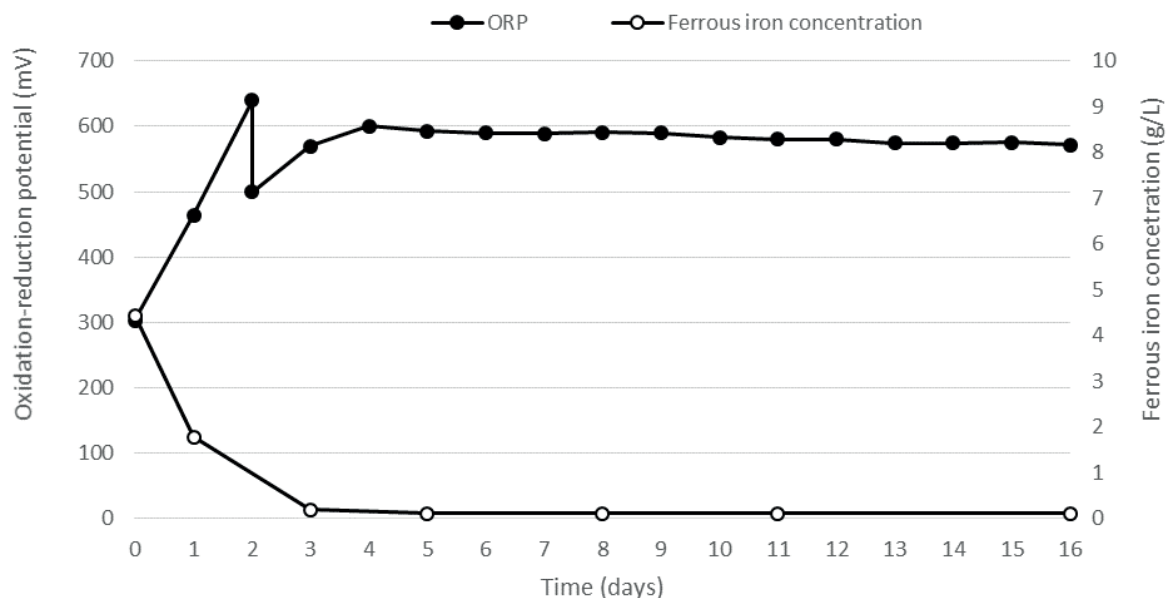


FIGURE 9: The graph indicates the pH and ORP values for different pulp densities. In each adaptation cycle, on day 2, when ORP reaches  $\geq 600$  mV, BOF material was added to the culture flask.





**FIGURE 10:** The graph indicates the ORP values (mV) and ferrous iron concentration (g/L) for 5% (w/v) pulp density at pH 1.5 over 16 days bioleaching. On the day 2, when ORP reaches  $\geq 600$  mV, BOF material was added to the culture flask.

170.8 mg/kg and Cu was 134 and 57.7 mg/kg according to ICP-MS and pXRF, respectively. After bioleaching, none of these metals were detected by pXRF, thus it was assumed that all the elements were dissolved or were below the limits of detection. Cr dissolution was only 2.7%. In terms of Fe and Bi, weight gain was detected after bioleaching. This suggests that these metals could not be recovered. This result is promising for further use of BOF material as a secondary iron resource as the iron did not leach. Further analysis is needed to determine the dissolution by ICP-MS. Further work is required to optimise bioleaching to improve metal dissolution and to understand the leaching kinetics.

#### 4. CONCLUSIONS

The characterisation experiments and literature review revealed that according to the carbon (<10%), iron (46.8%), zinc (1.2%) and lead (0.2%) content, the samples can be characterised as BOF sludge and dust. Negative LOI values indicated that weight gain can be an indication for a high iron content. Both ICP-MS analysis and p-XRF results verified the high Fe content.

Preliminary bioleaching experiments consisting of acclimating *A. ferrooxidans* are encouraging as the strain has been successfully adapted to 5% sludge and dust material. Bioleaching was performed at 5% pulp density with 22 g/L energy source and 10% (v/v) inoculum at pH 1.5. After bioleaching, 100% of Ti and Cu, 32% of Zn and 24% of Mn was removed. Higher bioleaching yield can be achieved by process optimisation. Fe and Bi did not dissolve. Due to iron not leaching, there is an opportunity to utilise BOF material as a secondary iron resource alongside metal recovery. Further work will be carried out to demonstrate and optimise such technological approach along chemical leaching.

This work has highlighted opportunities in metal recovery

from PMSD and the necessity to develop rapid assessment tools to aid in site characterisation for risk assessment and appraising resource opportunities.

The REGENERATIS project will harmonise existing databases from across North West Europe and develop an open-access platform to enable new business opportunities for metal recovery from PMSDs. Using the database and new data generated through the project, an open-source 4D SMART tool (SMARTIX) based on an artificial intelligence algorithm will be produced.

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**TABLE 2:** Table shows the p-XRF results of BOF sludge and dust and before and after bioleaching and the bioleaching yield. When elements could not be detected (NA) after bioleaching it was assumed all the element dissolved.

	Ti	Cr	Mn	Fe	Cu	Zn	Bi
BOF sludge and dust (mg/kg)	170.8	105.7	5960	432634.9	57.7	12742.8	348.7
Bioleaching residue (mg/kg)	NA	102.8	4539.4	481862.5	NA	8656.4	354
Bioleaching yield (%)	100	2.7	23.9	0	100	32.1	0

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