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Recycling Zinc from Alkaline batteries

Recovering Zinc from 1,5V AA alkaline batteries through hydrometallurgical techniques

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Chalmers University of Technology, 2017

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Abstract

Alkaline batteries are a type of primary batteries, accounting for ~80 % of batteries collected for recycling by the Swedish recycling company Renova. Renova also manages fly ash deriving from municipal solid waste incineration, which share a high zinc composition with black mass from alkaline batteries. Previous studies have been performed by Renova in order to extract zinc from fly ash through hydrometallurgy. This is a technique which has shown great potential and in this study, the same methodology as in the fly ash project was applied on black mass deriving from 1,5V AA alkaline batteries. This study examined if zinc can be recycled through a similar hydrometallurgical method as the fly ash, and if these processes could potentially be combined.

Different leaching parameters and their effects on zinc recovery from black mass were studied. The examined parameters were leaching media concentration, liquid-solid ratio and leaching time. Hydrochloric acid was used as leaching media, and the metal separation process consisted of zinc hydroxide precipitation by adding sodium hydroxide. This project was performed in a laboratory scale, with no economics aspects taken into consideration. The study was executed at Chalmers University of Technology, at the department of Civil and Environmental Engineering in collaboration with Renova.

This report concluded that the hydrometallurgical approach used on fly ash to extract Zn can, with slightly different parameters, be applied on black mass deriving from 1,5 V AA alkaline batteries. A leaching time of 30 minutes, 2 M hydrochloric acid as the leaching media and a liquid-solid ratio of 7 mLg^{-1}, resulted in a >95 % recovery of Zn in the leaching process and a total Zn recycling of 85 % was achieved.

KEYWORDS: Zinc recycling, Alkaline batteries, Black mass, Hydrometallurgy, Hydrochloric acid, Acidic leaching, Precipitation

Acknowledgements

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1 Introduction

With the rapid growth of portable electronical devices, both the need for and use of batteries has increased [1, 2, 3]. Alkaline batteries are a type of battery that is used globally and represent 51 % of the European portable battery market [4].

Alkaline batteries contain elements such as zinc (Zn) and manganese (Mn) as active substances, which supply the equipment with energy. The use of Zn is not limited to battery systems and is also used in numerous other products such as cosmetics, electronics, healthcare, and automobiles. Due to its extensive applicability, the demand and usage for Zn has increased in recent years and is currently higher than the accessibility, thus making it vital to recover the metal [5].

Alkaline batteries contain high amounts of Zn and since the metal availability is limited, recovery of Zn from spent batteries is highly relevant. According to David Dalek, a representative from the Swedish recycling company Renova, 1 200 ton batteries are processed yearly within the company and ~80 % of these are alkaline batteries. As the recovery of Zn is important, an effective recycling method is needed for these alkaline batteries.

There have been successful studies of extracting Zn from fly ash, originating from municipal solid waste incineration, through acid leaching. Since fly ash and alkaline batteries share a high Zn composition, it may be possible to recover Zn from alkaline batteries using the same hydrometallurgical approach. During the fly ash project, a pilot industrial unit was built to examine the possibility of separating different metals, with Zn as its prior concern. In this industrial unit, process water was used as leaching media, which is comparable to 1M HCl [6]. The most beneficial parameters concluded in the fly ash project, were a 30-min leaching time, liquid-solid ratio of 3 mLg^{-1} and a leaching media concentration around 1 M. As Renova manage both fly ash and alkaline batteries, it is appealing to examine if Zn can be recovered with the same methodology as the fly ash project from alkaline batteries [6].

1.1 Aim

The aim of this project was to examine the applicability of acid leaching on black mass deriving from 1.5V AA batteries to extract Zn, using a hydrometallurgical approach. The applied recycling technique consisted of acidic leaching of black mass followed by precipitation with sodium hydroxide (NaOH) to produce a zinc hydroxide ($Zn(OH)_2$) precipitation. This project has been performed at Chalmers University of Technology in collaboration with Renova.

1.2 Clarification of the aim

In this study, the efficiency of the Zn recycling from both the leaching and precipitation steps were analyzed, as well as the composition of the samples. The amount of recovered Zn from the whole process has also been determined. The problems that have been examined during this project are:

- *Can Zn be recovered from 1.5V AA alkaline batteries using a hydrometallurgical method?*
- *How will the leaching time, liquid-solid ratio and leaching media concentration affect the efficiency of the leaching and degree of Zn recycling?*
- *How does the optimal parameters from this study compare to the ones used for the fly ash project?*

The influence of the following parameters was examined to see how they affect the amount of extracted Zn from crushed alkaline batteries:

- Concentration of hydrochloric acid (HCl) (leaching media)

- Liquid-solid ratio (L/S)
- Leaching time

Since there is a desire to somewhat combine the recovery process of crushed alkaline batteries with the recycling process of fly ash, this project has also examined if similar parameters can be used.

1.3 Limitations

This study examined crushed 1,5V AA alkaline batteries with the plastic, paper and metal cover removed, black mass, as starting material. This study only investigated if Zn could be recovered through acid leaching in a laboratory scale, where no economic aspects are taken into consideration.

2 Background

2.1 Batteries

A battery is an electrochemical device that powers appliances through conversion of chemical energy to electrical energy [4, 7]. Depending on the characteristics, batteries are divided into two groups: primary and secondary. A big difference between these categories is that primary batteries will lose their ability to produce electrical energy after discharge while the secondary batteries are rechargeable and thereby having a longer lifespan [8, 9].

With the varying energy demand of different equipment, it is important that batteries have the ability to sustain them for longer periods. Altering the composition of the electrodes and electrolytes will give each battery system specific characteristics and thereby making it possible to meet the energy requirement for various devices. The main difference between different battery systems are changes in the battery's interior composition, which will change depending on e.g. how much energy the battery needs to supply to a specific equipment [4, 7].

Alkaline batteries are primary batteries and the general structure of the battery system is illustrated in Figure 1.

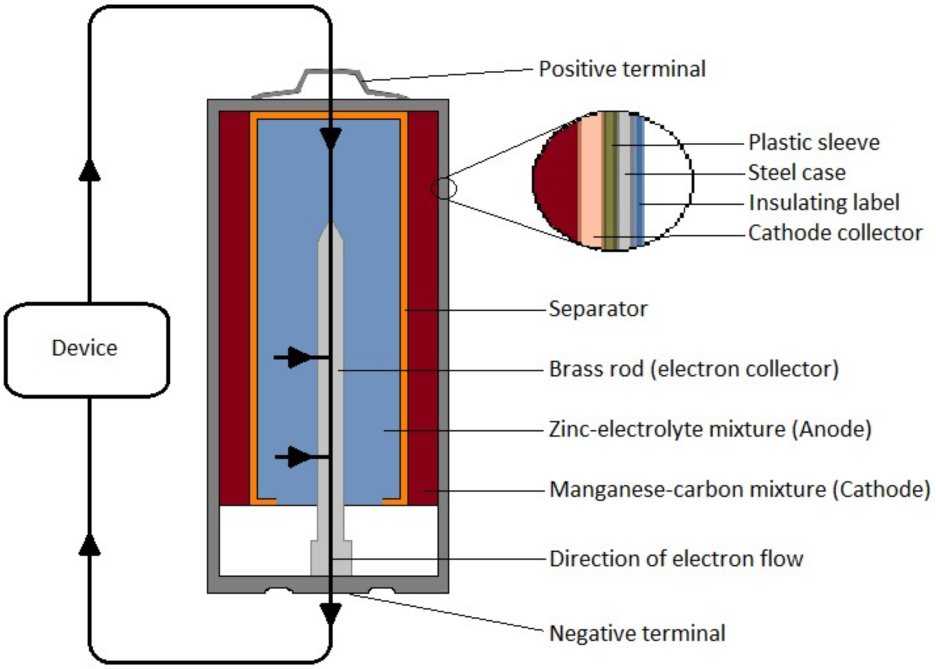

Figure 1: An alkaline-manganese battery system and its different components and structure

An alkaline battery system is divided into two cells, an anode and cathode cell. The anode, illustrated by the blue section in Figure 1, is composited by a zinc powder mixture combined with a potassium hydroxide (KOH) electrolyte. The counter part to the anode cell is the cathode cell, represented by the red section in Figure 1, and it consists of a mixture of manganese dioxide (MnO_2) and carbon (C). To enable the battery to produce electrical energy, a separator is inserted between the cells. This will cause a charge separation among the two electrodes, hence allowing the battery to generate electric energy. To achieve a functional electron flow in the battery, a brass rod is connected to the anode mixture. This rod collects and transports the produced electrons toward the negative terminal [4, 10]. The overall reaction scheme for an alkaline-manganese battery cell is described in Reaction 1 [10].

$$Zn + 2Mn\ \ _2 \rightarrow ZnO + Mn_2O_3 \qquad (1)$$

As mentioned, the alkaline battery composition consists of several different metals. However, they generally do not present as pure metals but as compounds with other elements [4, 10]. It is difficult to use regular separating methods to isolate Zn from battery mixtures. Conversional extraction of Zn from ores often uses zinc sulfide (ZnS) that is converted to zincite (ZnO) through roasting. ZnO is then leached with nitric acid (HNO_3) to obtain a Zn solution. Contaminations in the leachate are subsequently removed to produce a pure Zn metal with e.g. electrolysis [6].

To recover Zn from alkaline batteries, various techniques can be exploited where each recycling process have different advantages.

2.2 Recycling process

There are different processes available used to recycle batteries; physical, pyrometallurgical and hydrometallurgical, illustrated in Figure 2 [4]. The first step in all recycling methods for batteries is the physical recovery process, where batteries are sorted according to their composition, as different batteries require different recycling techniques due to the variation in chemical structure [11]. According to Dalek, Renova currently utilize a physical recycling process, where 1,5V AA alkaline batteries are separated and subsequently crushed. The iron cover, paper and plastics are removed from the graphite and metallic oxides using magnets and stratification, after which they are recycled [11]. The remaining components, black mass, can then managed through various techniques.

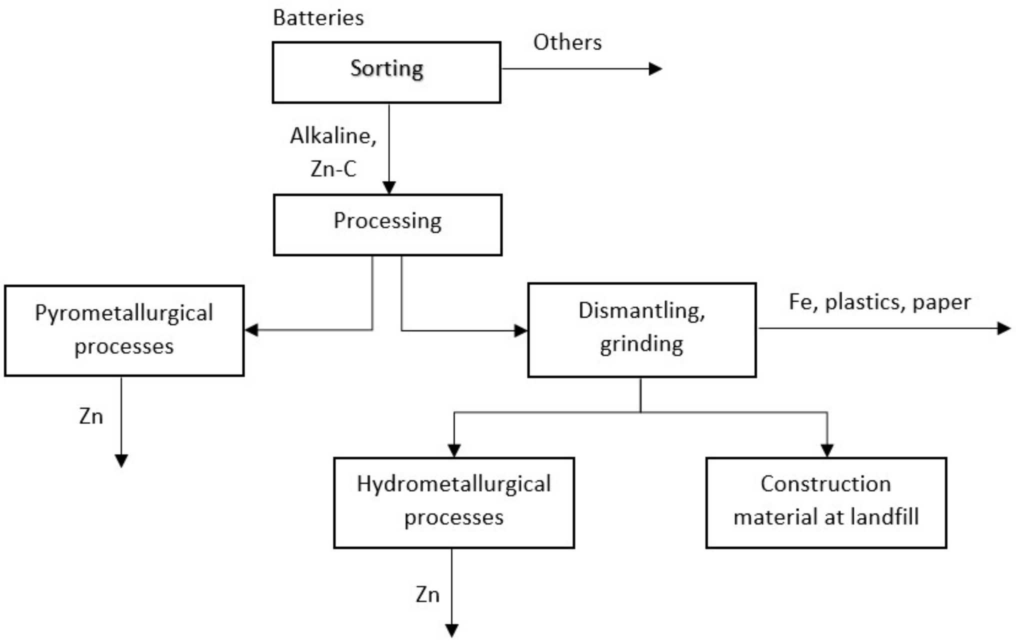

Figure 2: Different disposal methods and recycling techniques of batteries

There are several disposal methods of black mass. One way is to use the black mass as a construction material at landfills. This method is currently utilized by Renova, according to Dalek. As alkaline batteries can contain various toxic elements such as mercury (Hg), stabilization, a method which aims to evade contact between metals and the environment at a landfill, was developed. It is not industrially widespread due to the associated cost [4].

Using black mass as a construction material is a strain on natural resources, as precious metals are lost. The black mass from alkaline batteries contains between 15 % and 25 % Zn alone [11, 12, 13, 14]. A 2012 analysis of the components of black mass deriving from 1,5V AA alkaline batteries from Renova, found in Appendix A, shows a content of 24% Zn and being able to harvest this metal is of high interest. With current depletion of high grade ZnS-ores and as the need for Zn increases, alternative sources of Zn are essential to sustain our way of life [5]. The black mass may, as earlier mentioned, also contain dangerous substances. This makes it unsuitable as a construction material, as it may pollute the environment [4, 15]. Another option is using pyrometallurgical or hydrometallurgical processes, or a combination of the two, to recover the metallic oxides from batteries [16].

2.2.1 Pyrometallurgical techniques

Pyrometallurgical processes are the most industrially widespread methods used to recycle batteries. These types of techniques are generally simple and do not require the dismantling of batteries. Pyrometallurgical methods selectively volatilize metals, followed by a condensation [11].

One of the industrially applied pyrometallurgical processes is the BATREC method from Switzerland. The BATREC process can be applied on all batteries, except for Cd-Ni. The initial step is to recover Hg through distillation. The batteries are heated to 1500°C, after which Zn is condensed as a metal or oxide. Mn and Fe can be retrieved as an alloy [14, 17, 18]. There are multiple other industrial methods, such as TERA and AFE VALDI. The AFE VALDI process recovers Zn and Mn, while the TERA technique also recovers Hg from alkaline batteries [18].

When recycling alkaline batteries through pyrometallurgy, Zn and Hg is fully recovered. However, dust collecting systems and gas cleaning systems are essential for these processes, as the techniques can cause both pollution through the dust created and emit gas to the environment. These types of techniques are also generally very energy consuming because of the high temperatures [11], all of which is why hydrometallurgical approaches are appealing.

2.2.2 Hydrometallurgical techniques

Another way of recycling metals in black mass from batteries is through hydrometallurgical processes. This recycling technique consists of leaching, followed by metal separation [16]. The metal separation can occur through electrolysis, extraction, or precipitation [17]. These methods commonly require pretreatment in the form of grinding, as it improves the efficiency of the leaching step [11]. An additional step can also be neutral leaching, i.e. washing, to remove salts from the black mass [19].

There are many hydrometallurgical processes that can be applied to recover Zn from spent alkaline batteries [14, 16, 20]. In 2012 Buzato et.al. studied both alkaline (6M,10gL^{-1}, 2h, 80°C) and sulfuric (2M, 100gL^{-1}, 1h, 80°C) leaching of Zn-Mn0$_2$ batteries in laboratory scale and concluded that 68% and 96% Zn respectively, was recovered. This establishes that both base and acid leaching could be applied in the extraction process of Zn from alkaline batteries [20].

Multiple studies have also been done using HCl as the leaching media, and the method is showing promise. In 2013 Cabral et.al used HCl to leach alkaline and saline batteries in laboratory scale, at 20°C and 80°C for 1 hour, using a liquid-solid ratio of 20 mLg^{-1}, and confirmed that Zn can effectively be leached even at lower temperatures [21]. Reaction 2 shows the chemical reaction in the leaching process using HCl as leaching media.

$$ZnO(s) + 2\,HCl \rightarrow\ ZnCl_2(aq) + H_2O \tag{2}$$

There are different ways of recovering metals from the leachate in a hydrometallurgical process; electrolysis, extraction, and precipitation. Precipitation has proven to be an efficient method for Zn separation in a process for alkaline batteries, since Zn precipitates at pH ~8 and Mn at pH ~10 [19]. It is also a relatively simple and industrially applicable method [18]. Different chemicals can be used for the pH adjustment required for the precipitation process, such as NaOH [22]. The reaction for a precipitation of Zn^{2+} to $Zn(OH)_2$ using NaOH is illustrated in reaction 3.

$$ZnCl_2(aq) + 2\,NaOH \rightarrow Zn(OH)_2(s) + 2\,NaCl \tag{3}$$

There are multiple industrially applied hydrometallurgical methods used to recycle alkaline batteries, such as Revabat. Revabat is used in Belgium, Luxemburg, and the Netherlands among others, and recovers zinc sulfate ($ZnSO_4$) and manganese sulfate ($ZnSO_4$) from alkaline and Zn-C batteries [18]. This process currently recycles more than 3 000 tonnes per year [23]. Another industrially applied hydrometallurgical technique used on alkaline batteries is MMM-Sedema, which is operating in Belgium and recovers Zn and MnO_2 [18]. These hydrometallurgical methods are not, as already mentioned, as widespread as pyrometallurgical recycling techniques [11].

Hydrometallurgical recycling of black mass is preferable to using it as a construction material at a landfill, as the metals are recycled and re-used, especially since Zn is a limited resource [5]. A hydrometallurgical approach has the potential to recover the leachate and that fact the hydrometallurgical process does not contribute to any air pollution, often makes hydrometallurgical recycling methods preferable to pyrometallurgical ones. The wastewater does, however, contribute to secondary pollution, a lot of chemicals are required and these procedures are also time consuming, relative to pyrometallurgical techniques [11].

3 Experimental

In this section, the method used in this study will be presented, along with the analytical methods.

3.1 Sample preparation

The black mass was dried in 105°C for 12 hours. The first two experiments used rough, black mass as seen in Figure 3.

Figure 3: Black mass from AA 1,5V alkaline batteries

As there were pieces of iron and plastics remaining in the mixture, these fragments were sorted out in experiments 3-9. The composition of rough and sorted black mass are found in Table 1. The main components that are found in the masses are K, Mn and Zn.

Table 1: Composition of black mass, both rough and sorted (analysis from 2017)

Element	Rough black mass (mg/kg)	Sorted black mass (mg/kg)
Si	<1000	<1000
Al	341	378
Ca	<500	<500
Fe	3 340	4 090
K	26 300	26 300
Mg	91	106
Mn	176 000	182 000
Na	<1000	1 090
P	<50	<50
Ti	274	316
As	<5	<5
Ba	835	661
Be	<1	<1
Cd	59.4	69.4
Co	53.5	62.5
Cr	<5	<5
Cu	220	251
Hg	7.34	10.2
Nb	<0.5	<0.5
Ni	700	888
Pb	73.7	76.6
S	1070	1 120
Sc	<0.2	<0.2
Sr	18.1	20
V	1.58	1.6
W	0.479	0.475
Y	1.62	1.93
Zn	127 000	131 000
Zr	<1	<1

3.2 Selecting parameters

HACH analysis of Zn was performed on the initial tests leachates to compare and decide the starting parameters for the concentration of HCl and liquid-solid ratio, see Appendix B. The leaching time for each trial run was set to 30 minutes. This analysis technique was also used to compare the Zn concentration in the filtrates after different pH adjustments to find an appropriate pH interval for the precipitation of $Zn(OH)_2$. HACH analysis was not used to obtain any quantitative results. The following starting parameters and pH adjustment after the leaching step were decided:

- 1 M leaching media concentration
- Liquid-solid ratio of 5 mLg^{-1}
- Leaching time 30 minutes
- pH adjustment interval 7-8

The parameters in each experiment will vary between:

- Leaching media (HCl) concentration: 0,5 M, 1 M and 2 M
- Liquid-solid ratio: 3, 5 and 7 mLg^{-1}
- Leaching time: 30, 60 and 120 minutes

3.3 Recycling Zn from batteries

The different experiments were performed according to the process scheme illustrated in Figure 4. Acid leaching was executed, for various amounts of time, to extract Zn^{2+} from black mass. The leachate was then separated from the battery mass after which sodium hydroxide was added to the solution to create a precipitation containing $Zn(OH)_2$.

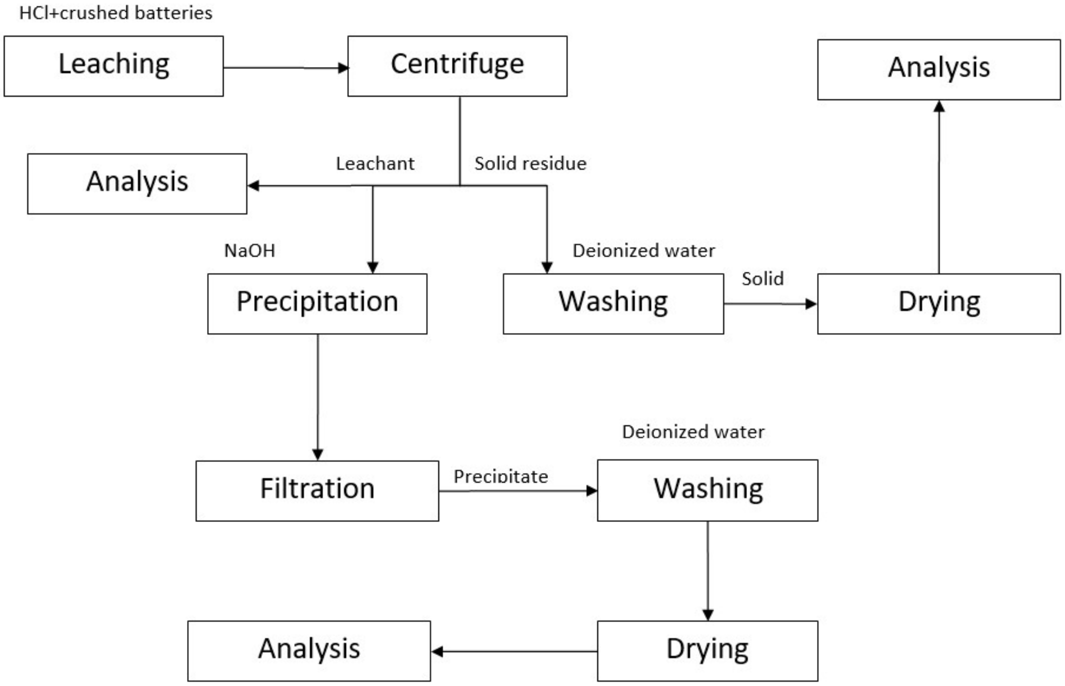

Figure 4: Overview of the experimental procedure

3.3.1 Leaching process

Black mass and HCl were added to a centrifuge tube and was placed on a shaking table (100 rpm) for a set amount of time. The used amount of black mass, HCl and leaching time are listed in Table 2 for each experiment. The leachate was then separated from the remaining battery mass through centrifugation and decantation. The residual black mass was washed with deionized water (10 mL) for 30 minutes on the shaking table and subsequently centrifuged and dried (105 °C). Triplicates were done in each trial to attain reliable results.

Table 2: Parameters used in each experiment

Experiment	**L/S (mLg^{-1})**	**[HCl] (M)**	**Leaching time (min)**
1	5	1	30
2	5	2	30
3	5	0.5	30
4	3	1	30
5	7	1	30
6	5	1	60
7	5	1	120
8	5	2	30
9	7	2	30

3.3.2 Zinc recovery

The leachate (20 mL) was placed in a beaker and pH measured. A pH adjustment was then done with NaOH and the mixture was stirred for at least 30 minutes. The formed $Zn(OH)_2$ crystals were separated from the fluid through suction filtration (1-2 μm pore size) and subsequently washed using ultrapure water. Lastly, the crystals were dried (50 °C) and weighed. Appendix C and D, shows a more detailed description of the pH values of each sample in the different stages mentioned above.

3.4 Analytical methods

3.4.1 HACH analysis

HACH analysis was performed on all initials tests and experiment 1-3, see Appendix B to predict the Zn concentration of the leachate. The results indicated by HACH were verified by using the standard addition method, seen in Figure 5. It can be seen that the values of the orange curve are much lower than the blue standard line. The orange curve contains both standard and sample solution and should therefore be higher than the blue standard line. This behavior shows that the interferences in the samples are too extensive for the utilization of this analysis method. Consequently, HACH was only used for qualitative analyses for the initials tests and first 3 experiments. The data given in Figure 5 is found in Appendix B.

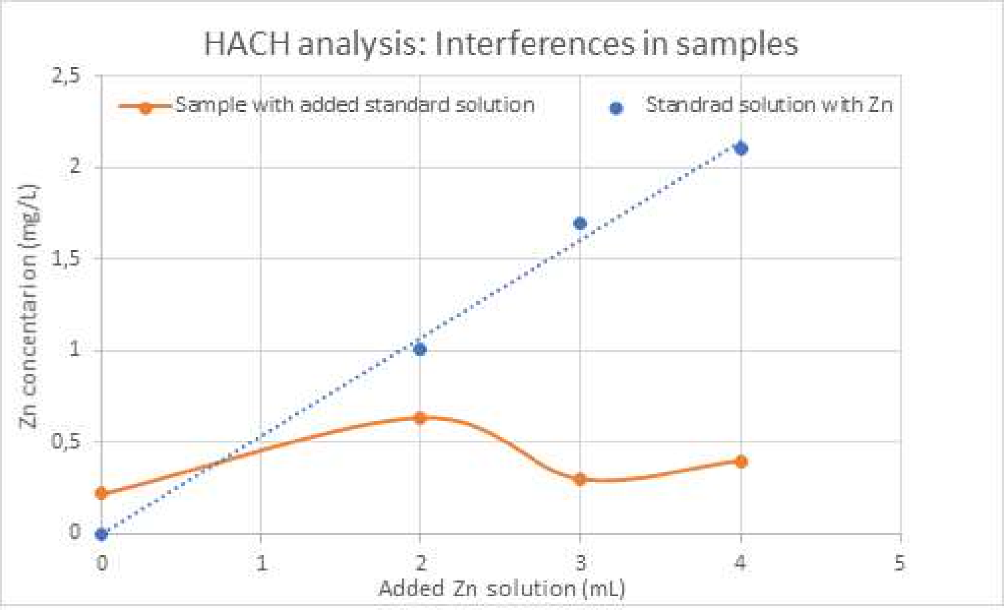

Figure 5: Standard addition method examining the accuracy of the HACH measurements and interferences of the samples

3.4.2 Inductively coupled plasma analysis

Inductively coupled plasma analysis (ICP) was used to analyze the contents of the dissolved precipitation samples. ICP uses plasma, containing charged particles, to ionize the sample. Both MS and SFMS analyze the substances in the sample according to their separate masses. The difference between them is that MS uses a high vacuum chamber to analyze elements, while SFMS utilizes magnetic and electrostatic sectors. With ICP-AES, the plasma will heat the sample so that the components will be excited and emit light that will be analyzed instead [24].

Analysis of leachate

All the leachate samples were sent to an external lab for analysis, and ICP-AES was used as the quantitative analysis method.

Preparation and analysis of precipitation

Each precipitation (0.2 g) was dissolved in HNO_3 (5 ml, 65%). The precipitations did not fully dissolve, see Figure 6. The samples were as a result centrifuged to separate the liquid and the solid. The compounds in the precipitation solution were analyzed with ICP-MS.

Figure 6: The centrifuged precipitation samples

Preparation and analysis of solids

The solids, listed underneath, were sent to and external lab and were analyzed with ICP-SFMS.

Six g of the following samples were analyzed with this technique:

- *Rough mass*
- *Sorted mass*
- Experiment 2 solid residue
- Experiment 5 solid residue
- Experiment 8 solid residue
- Experiment 9 solid residue

The solid residues in samples 2, 5, 8 and 9 were mixed together within each trial so that a representative average value could be determined. It should be noted that during the analysis of the masses ~0.2 g of the material did not dissolve except for experiment 2 were the undissolved mass were 0.35 g and were therefore not analyzed.

4 Results and discussion

4.1 Leaching process

In Figure 7, the average composition of the leachates from the triplicated experiments are found, expressed in mg element in leachate per kg black mass. The exact values are found in Appendix F, and the calculations are found in Appendix J under *Leaching.* For the experimental condition of each sample see Table 2.

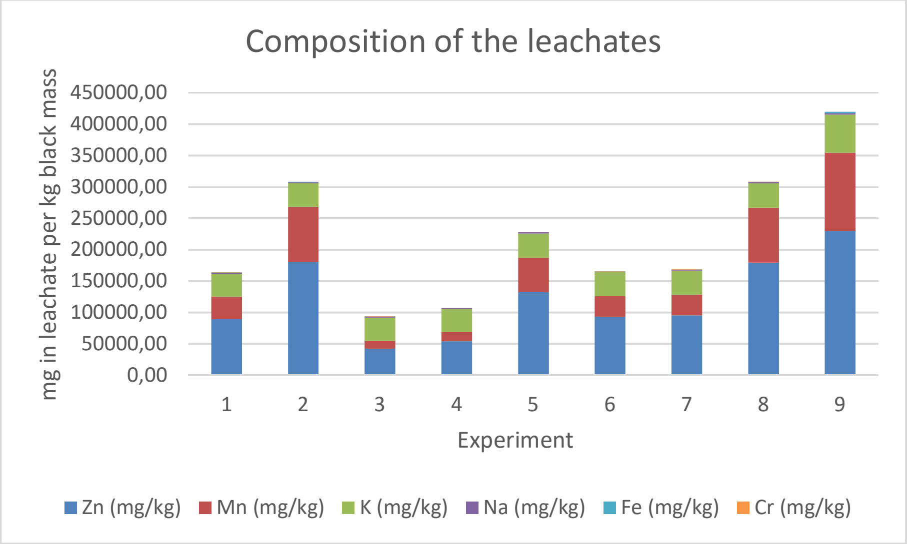

Figure 7: Composition of metals in the leachates

The leached amounts of Zn and Mn were affected by the tested parameters, as they vary from experiment to experiment. However, K, Na, Fe and Cr seemed to be relatively constant with the exception of experiment 9, where more of each element is recovered. This implies that it is possible to retrieve more of each element present in the black mass by adjusting leaching time, leaching media concentration and liquid-solid ratio. From Figure 7 it can be seen that experiment 9 has the highest amount of recovered Zn. This shows that the most favorable parameters were with a 30-min leaching time, liquid-solid ratio of 7 mLg^{-1} and a leaching media concentration of 2 M.

It is also notable that experiment 2 contained more than 10 times the amount of Fe compared to experiment 8, where the only difference was whether rough or sorted black mass was used. This indicates that the rough mass contained more Fe that subsequently was leached in the leaching process, a conclusion supported by the elevated levels of Fe also present the leachates from experiment 1.

Figure 8 illustrates how the recovered Zn in the leaching process correlated to the black mass which dissolved during leaching, calculations are found in Appendix J under *Leaching*.

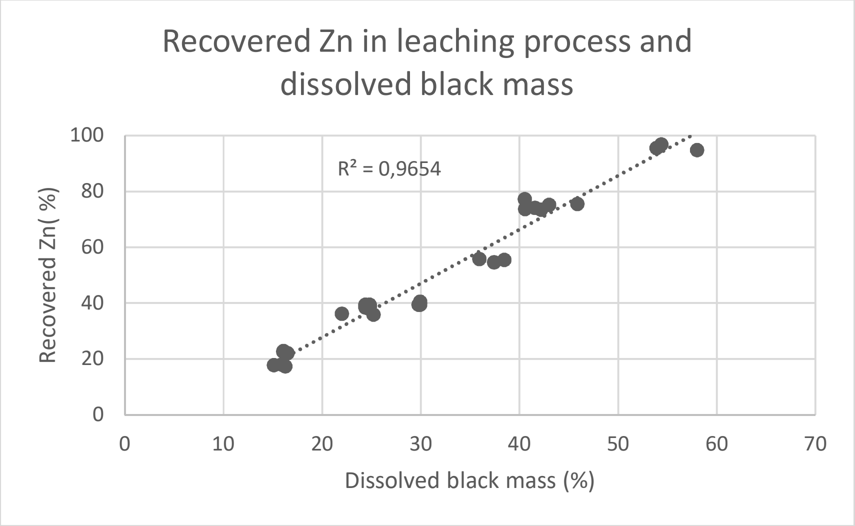

Figure 8: Correlation of recovered Zn with dissolved black mass

As seen in Figure 8, dissolved black mass and recovered Zn have a close to linear relationship. This confirms that recovered Zn increases when more black mass is dissolved. In some cases, the dissolution increases whereas the recovered Zn remains constant. This suggests that there are some samples where a higher concentration of components other than Zn have been leached into the solution. Figure 9 displays the relationship between recovered Zn and recovered Mn.

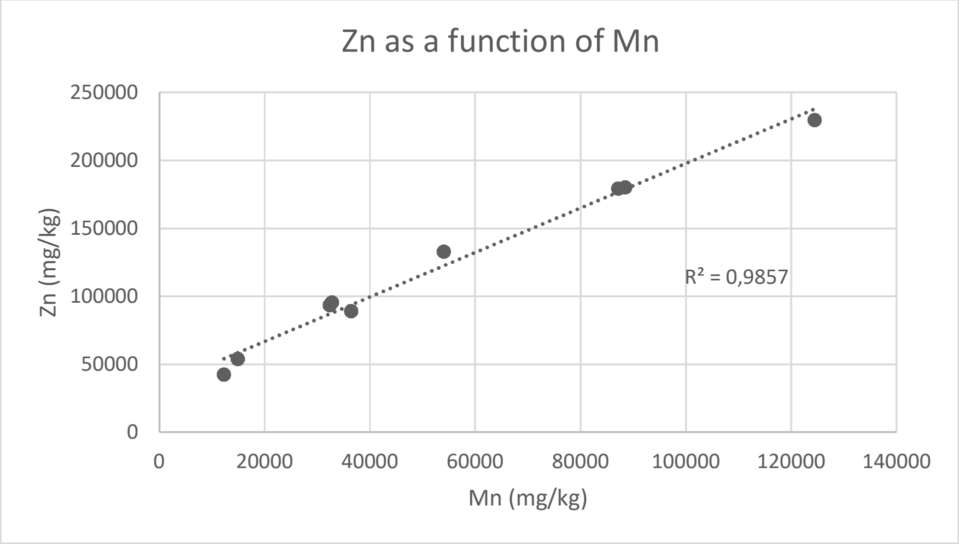

Figure 9: Zn as a function of Mn

As seen in Figure 9, there was a relationship between leached Zn and Mn during the experiments. This indicates that there was a correlation between the leaching of other components, such as Mn, and Zn. This claim is supported by the increase of leached components in experiment 9, where the highest percentage of Zn was recovered.

Table 3 presents the recovered Zn from black mass in the leaching step, both based on a 2017 analysis of black mass and an analysis Renova performed 2012, found in Table 1 and Appendix A respectively.

Table 3: Recovered Zn from black mass during leaching, based on both a 2017 and 2012 analysis of black mass

Experiment	Recovered Zn based on 2017 analysis of black mass (%)	Recovered Zn based on 2012 analysis of black mass (%)
1	70	37
2	142	75
3	33	18
4	41	23
5	101	55
6	71	39
7	73	40
8	137	75
9	175	96

As seen in Table 3, the recovered Zn exceeded 100% when the calculations were based on the 2017 analysis of black mass. This indicates that the 2017 analysis was inaccurate, either because of an error during analysis or because the sample tested was not homogeneous and therefore not representative of the black mass used. The Zn composition of the leached black mass from experiment 2, 5, 8 and 9 were examined and by using the known Zn compositions of the correlating leachates, the average Zn composition of the starting black mass was calculated, found in Appendix J under *Leaching.* The Zn composition of the original black mass was found to be 24%, identical to the 24% found in the 2012 analysis, exact values are found in Appendix C. Furthermore, Renova has not changed the way black mass is handled since 2012, according to Dalek, and consequently the calculations of the recovered Zn from the leachates were based on the 2012 analysis.

Figure 10 displays how recovered Zn correlated to leaching time. The leaching media concentration (1 M) and liquid-solid ratio (5 mLg^{-1}) remained constant and the leaching time differed between 30, 60 and 120 min.

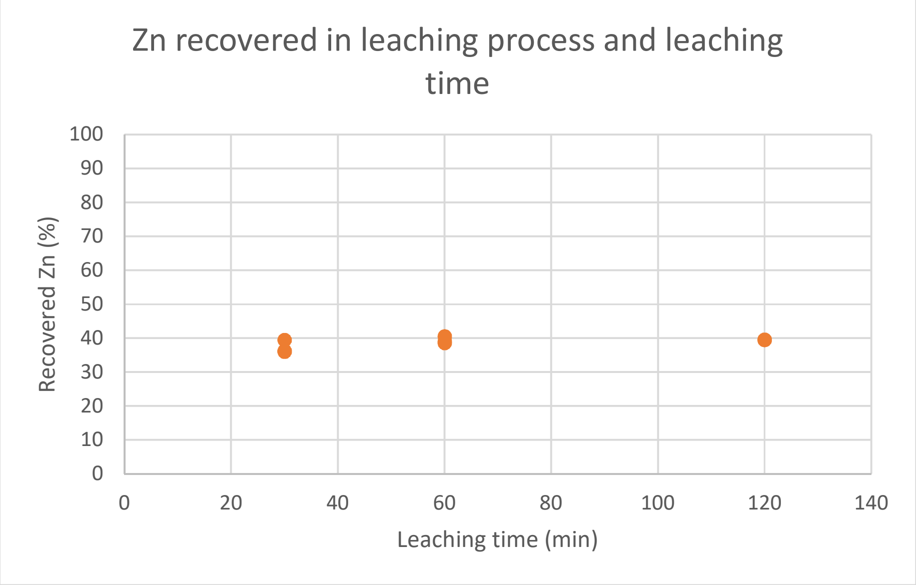

Figure 10: Recovered Zn as a function of leaching time

As Figure 10 illustrates, the recovered Zn did not notably increase with a longer leaching time. This indicates that an optimal leaching time is 30 min or shorter, as there virtually is no difference of recovered Zn between 30 min or 4 times as long, 120 min.

Figure 11 shows the relationship between recovered Zn and liquid-solid ratio. Leaching media concentration (1 M) and leaching time (30 min) remained constant, while liquid-solid ratio varied between 3, 5 and 7 mLg^{-1}.

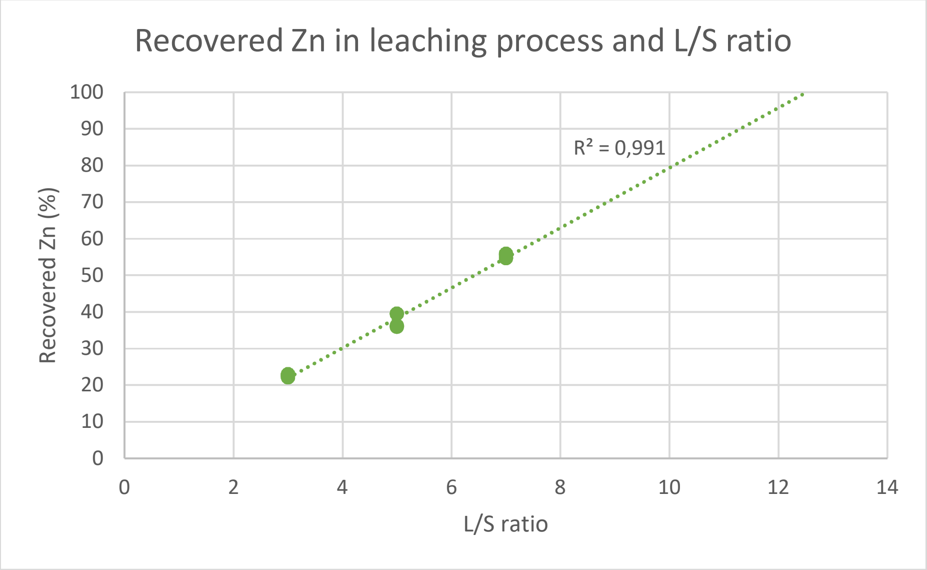

Figure 11: How recovered Zn correlates with liquid-solid ratio

There is a linear relationship between recovered Zn and liquid-solid ratio, where a higher ratio recovers more Zn from the black mass, seen in Figure 11. When further examining this relationship, the recovered Zn reaches 100 % with a liquid-solid ratio of ~12.5 mLg^{-1}.

How recovered Zn from black mass correlated with leaching media concentration is illustrated in Figure 12. The liquid-solid ratio (5 mLg^{-1}) and leaching time (30 min) was constant and the leaching media concentration varied between 0.5, 1 and 2 M.

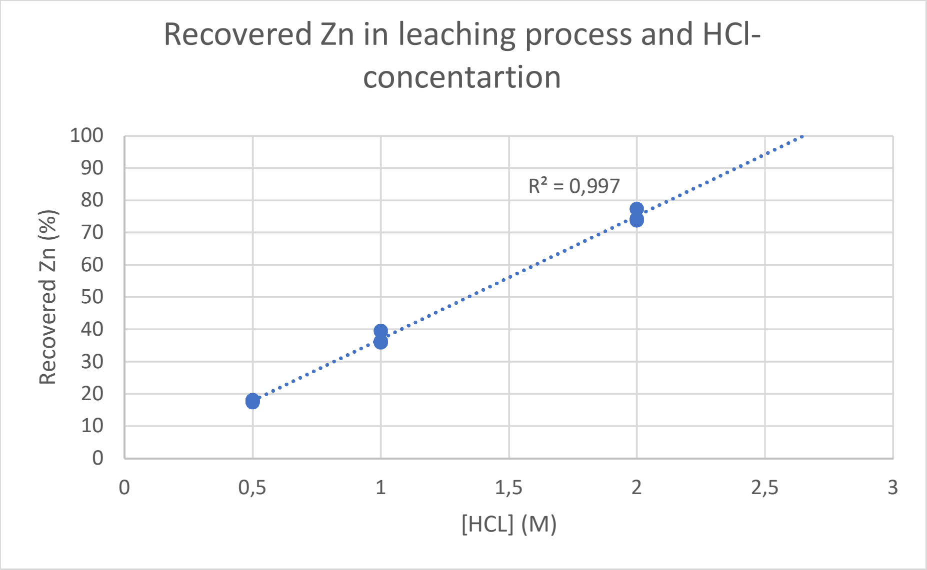

Figure 12: Recovered Zn as a function of leaching media concentration

The recovered Zn and leaching media concentration was proportional, where a higher concentration resulted in a higher percentage recovered Zn from black mass during leaching, displayed in Figure 12.

Extrapolating this linear relationship suggest that when a liquid-solid ratio of 5 mLg^{-1} is used, a HCl concentration of 2.6 M recovers 100 % of the Zn present in black mass.

As already mentioned, the efficiency of the leaching process was greatly affected by both leaching media concentration and liquid-solid ratio, and the results suggests that a higher HCl concentration and liquid-solid ratio recovers more Zn. Figure 13 displays the relationship between recovered Zn and liquid-solid ratio, where 2 M HCl was used as leaching media with a leaching time of 30 min.

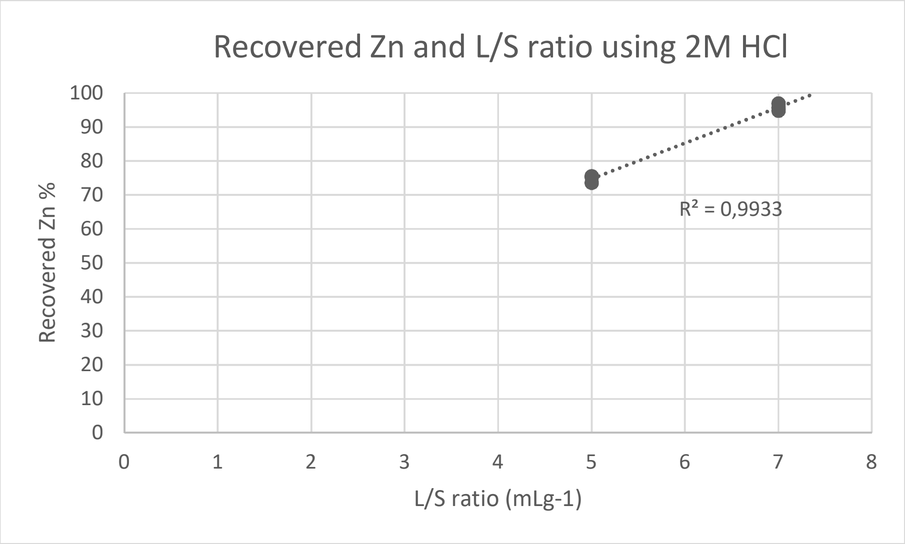

Figure 13: Extracted Zn and L/S ratio when 2M HCl was used as the leaching media, with a leaching time of 30 min

As seen in Figure 13, the recovered Zn increased from 75 % to 96 % when the liquid-solid ratio increased from 5 mLg^{-1} to 7 mLg^{-1}. Under these conditions, 100% of the Zn in black mass can be recovered when a liquid-solid ratio of 7.4 mLg^{-1} is used. Comparing these results to those where 1M HCl was used, the recovered Zn increased from 37 % to 75 % and from 55 % to 96 % for 5mLg^{-1} and 7 mLg^{-1} respectively. This supports the earlier claim that the leaching media concentration and liquid-solid ratio both improve the recovery of Zn when increased.

4.2 Metal separation process

The metal separation process consisted of a precipitation method where NaOH was added, done to isolate Zn from the leachate. For the analysis, these precipitations were dissolved in HNO_3 (65 %), where an average of 22 % of each precipitation remained solid. The analysis is found in Appendix G. In Table 4 the Zn and Zn calculated as $Zn(OH)_2$ present in the precipitations are displayed. The $Zn(OH)_2$ percentage was based on the assumptions that 100 % of Zn in the analysis of the dissolved precipitations were $Zn(OH)_2$ when solid and that all Zn dissolved, the calculations are found in Appendix J under *Precipitations.*

Table 4: Average value of Zn and $Zn(OH)_2$ in precipitations

Experiment	**Zn (%)**	**$Zn(OH)_2$ (%)**
1	49	75
2	55	84
3	55	84
4	50	76
5	51	78
6	49	74
7	51	78
8	49	75
9	49	75

As seen in Table 4, all precipitations had a similar Zn composition, where the average was 51 % Zn. A more detailed table of the Zn composition is found in Appendix D. They thereby had a purity of 77 %, as all Zn in the precipitations presumably were found as $Zn(OH)_2$ in solid form, according to Reaction 3. This indicates that the different parameters had no impact on the Zn composition of the precipitations. Furthermore, this also suggests that the Zn concentration in the leachate had no influence on the Zn composition of the precipitations. Some precipitations, such as sample 3.3, have a total composition that adds up to more than 100 %, see Appendix D, which suggests that some contaminations are present.

To further explore why the precipitations were partly insoluble in HNO_3 (65%), both pH of the leachate and pH after precipitation and their relationship to insoluble precipitation were examined. Figure 14 shows the correlation between insoluble precipitation and pH.

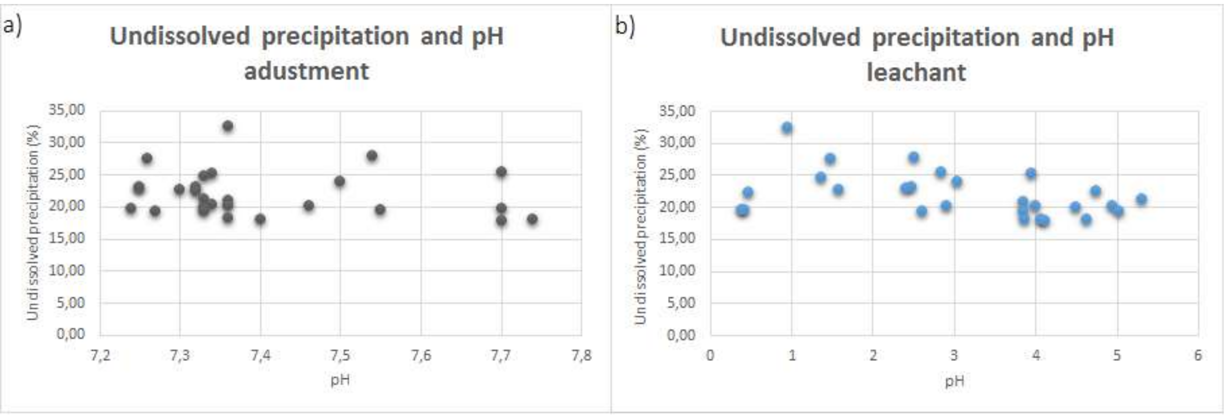

Figure 14: The undissolved precipitation and a) pH adjustment and b) pH of the leachate

As illustrated in Figure 14a), there was seemingly no relationship between pH after the precipitation process and insoluble precipitation, yet there seemed to be slight correlation to the pH of the leachate, seen in Figure 14b). As there were irregularities within the results, no conclusion could be drawn.

There was a higher percentage of insoluble precipitation in experiment 2, and this might derive from the fact that the samples were not homogeneous and that different, insoluble compounds have been leached and precipitated. This is supported by the fact that rough mass was used in experiment 2, as rough mass is more heterogeneous due to the iron and plastics present. The insoluble precipitations have not been analyzed, but based on the analysis of the dissolved precipitation, leachate and some of the liquids remaining after precipitation, filtrate, it is plausible that substances like Fe, Cr and Mn have created insoluble complexes during pH adjustment and drying.

As already mentioned, 22 % of all precipitations remained solid and therefore the complete composition of all precipitations has not been determined. However, the filtrates from experiment 2, 5, 8 and 9 were examined, found in Appendix H. By comparing the filtrate analysis with that of the leachate, the compositions of the precipitations were established. In Figure 15 the average compositions of the triplicated experiments are found. It is notable that experiment 2 and 8 are duplicates, and differs only in whether rough or sorted black mass was used.

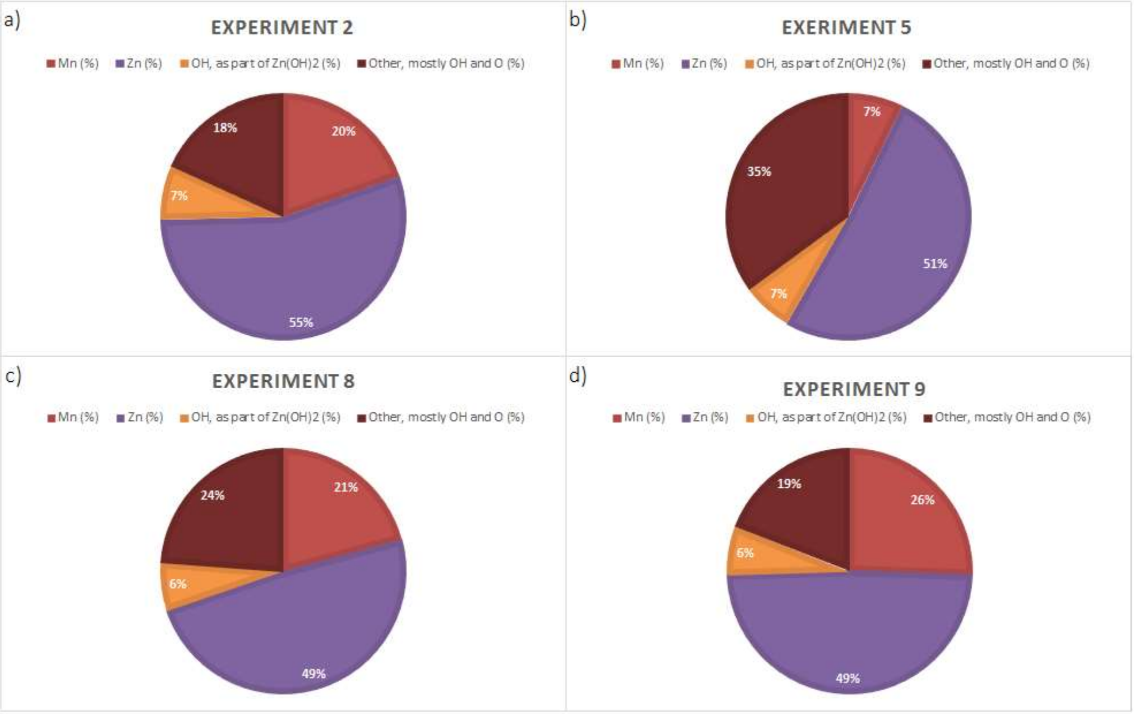

Figure 15: Compositions of the precipitations from experiment a) 2, b) 5, c) 8 and d) 9

As mentioned, all precipitations consist of ~50% Zn, represented by purple in Figure 15. The $Zn(OH)_2$ is represented by both purple and yellow, as Zn appears as a $Zn(OH)_2$ compound after precipitation. Mn is illustrated by light red and the maroon represents other components, such as Fe, Cr but mostly OH and O, as all precipitated components presumably reacted with the OH group in NaOH. As seen in Figure 15 the domain of these two groups vary from experiment to experiment. This suggests that the different parameters affect the remaining composition of the precipitation, having no effect on $Zn(OH)_2$.

Most precipitations did not have a pure white color, as $Zn(OH)_2$ has, rather different shades of white. The color variation amongst the precipitations can be seen in Figure 16.

Figure 16: The different precipitations, (from the left) experiment 1 to 9

As Figure 16 illustrates, samples from experiment 9 all have a darker color than the rest. These samples also contained exponentially more Fe, ~0.5% compared to the average of 0.03%. The irregular discoloration may therefore derive from different Fe- complexes formed during the pH adjustment. The variety in coloration between the samples probably stems from different precipitated compounds, a claim supported by the difference in composition amongst the precipitations.

The average Zn precipitation, the amount of Zn that precipitated from the leachate during precipitation, is shown in Table 5, as well as the pH adjustment made during precipitation. The exact values from each experiment are found in Appendix D.

Table 5: Zn precipitated from leachate and pH adjustment

Experiment	Zn precipitated (%)	pH adjustment
1	74	7,30
2	89	7,32
3	83	7,30
4	88	7,34
5	88	7,55
6	88	7,34
7	88	7,60
8	89	7,47
9	89	7,42

In experiment 1, the percentage of Zn precipitated from the leachate is notably lower than the rest. Some circumstance caused a repressed precipitation of Zn in these samples. As seen in Table 5, most experiments have similar amounts of Zn precipitated when the pH adjustments were between 7,3-7,6. A plausible source the dissimilarity may be irregularities in the pH meter due to inadequate calibrations. Consequently, the precipitation of Zn may have been inhibited if the pH adjustment was lower than the indicated value.

In two cases, sample 2.1 and 3.3, the precipitated Zn exceeded 100 %. This is likely because of different contaminations and these values have been excluded when calculating the average of Zn that precipitated from the leachates.

Apart from these irregularities, the process precipitated an average of 88 % of the Zn present in the leachates, in all experiments. This indicates that the precipitation process is neither affected by leaching time, liquid-solid ratio nor leaching media concentration.

4.3 General discussion

The leaching process was affected by the liquid-solid ratio and leaching media concentration, where increased values leach more Zn. These parameters affected the pH of the leachate, and Figure 17 illustrates the relationship between the Zn recovered form black mass during leaching and pH of leachate.

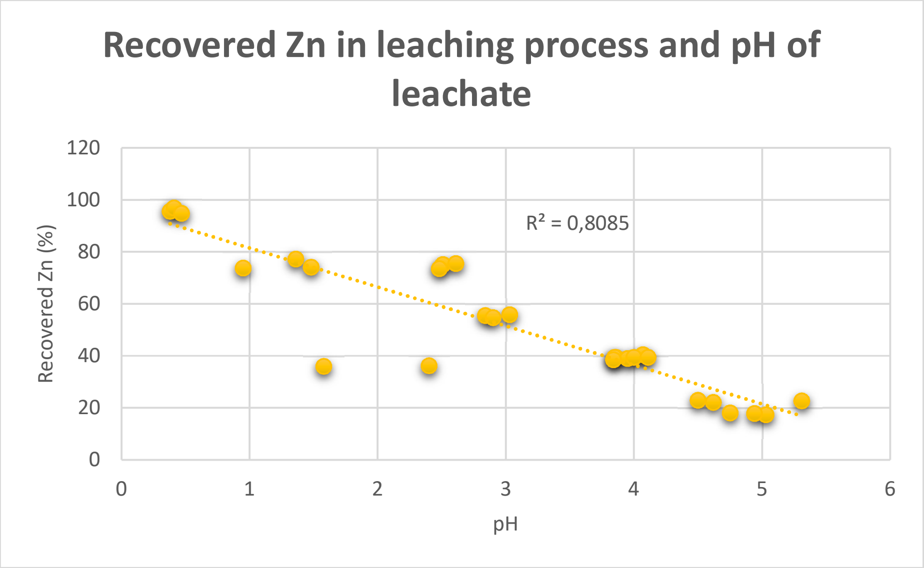

Figure 17: Recovered Zn from black mass as a function of pH in leachate

There are some irregularities, yet a clear correlation can be observed. This leads to the conclusion that a lower pH in the leachate leaches more Zn from the black mass.

There were some variety in the results between the triplicates. In experiment 3-9, the results were more evenly distributed, regarding both recovered Zn and pH of leachate, compared to experiment 1 and 2. In experiment 1 and 2 rough mass was selected and this have affected the distribution of the test results, as substances in rough black mass is more unevenly distributed compared to sorted black mass.

There is a ~10% margin of error in the ICP analysis methods, which may explain some of the abnormalities in the results. Furthermore, the experiments were performed in room temperature, which may differ from day to day. This may also have influenced the results but since the difference is marginal it is unlikely that this were the cause of any big variations.

The precipitation process had, as stated, a relatively constant precipitation of Zn with an average of 88 %. To improve the precipitation process, an increased pH interval should perhaps be applied during the pH adjustment. There are also other components, such as Fe- and Mn complexes present in the precipitations. The precipitation process is only effective if these complexes easily can be separated from Zn.

The degree of recycling, the percentage of Zn from the black mass present in the precipitation, is a measurement of how effective the whole recycling process was regarding Zn. The degree of recycling from all experiments is found in Table 6. The calculations are found in Appendix J under *Full process* and the exact values are found in Appendix E.

Table 6: Degree of recycling in all experiments, the percentage of Zn from black mass present in the precipitation

Experiment	**Degree of recycling (%)**
1	28
2	74
3	16
4	20
5	48
6	34
7	35
8	67
9	85

The degree of recycled Zn was rather low for most experiments, and was highest in experiment 9. As the precipitation had a similar percentage of precipitated Zn in all experiments, the leaching process mostly affected the degree of recycled Zn.

5 Conclusion

This hydrometallurgical approach can be applied to recycle Zn from black mass deriving from 1,5V AA batteries. The leaching time had no impact on the recovery of Zn within the studied interval, 30 to 120 min. However, both liquid-solid ratio and leaching media concentration affected the recovery greatly, where higher values increases the Zn recovery. These two parameters both affected the pH of the leachate, where higher values decrease the pH. This leads to the conclusion that a lower pH of the leachate allows for more Zn to be leached from black mass, thereby increasing the recycling of Zn.

The metal separation process, $Zn(OH)_2$ precipitation by adding NaOH, precipitated an average of 88 % of Zn found in the leachates and produced a precipitation containing 77 % $Zn(OH)_2$. This process was unaffected by the different leaching parameters, and seemed to depend on pH adjustment. To recover more Zn from this process, a higher pH interval for the pH adjustment should be explored. Since the precipitation contains 23 % components other than $Zn(OH)_2$, the possibility of separating Zn should also be examined.

In this project, a leaching time of 30 min, a liquid-solid ratio of 7 mLg^{-1} and a leaching media concentration of 2 M proved most effective and recycled 85 % Zn from black mass, with a 96 % recovery of Zn in the leaching process and a 74 % $Zn(OH)_2$ composition of the precipitation. Compared to the fly ash project, the same leaching time was used, yet more than a doubled liquid-solid ratio and the doubled leaching media concentration was needed to achieve similar results. If the leaching media concentration from the fly ash project were to be applied to the black mass, the liquid-solid ratio would have to increase to 12.5 to attain a similar outcome.

5.1 Future work

For future work, this method of Zn recycling should be evaluated in a larger scale. Studying Zn recycling through hydrometallurgy using different temperatures should also be examined further, to investigate if a similar trend as with liquid-sold ratio and leaching media concentration can be observed. Different leaching agents, number of leaching steps and various metal separation processes should also be explored, as well as including different reductants in the process. Exploring the Mn recovery is also of high interest, as black mass also has a high Mn composition. The economic aspects of this process should also be examined, as the potential for financial success often determines whether a process like this would be implemented industrially.

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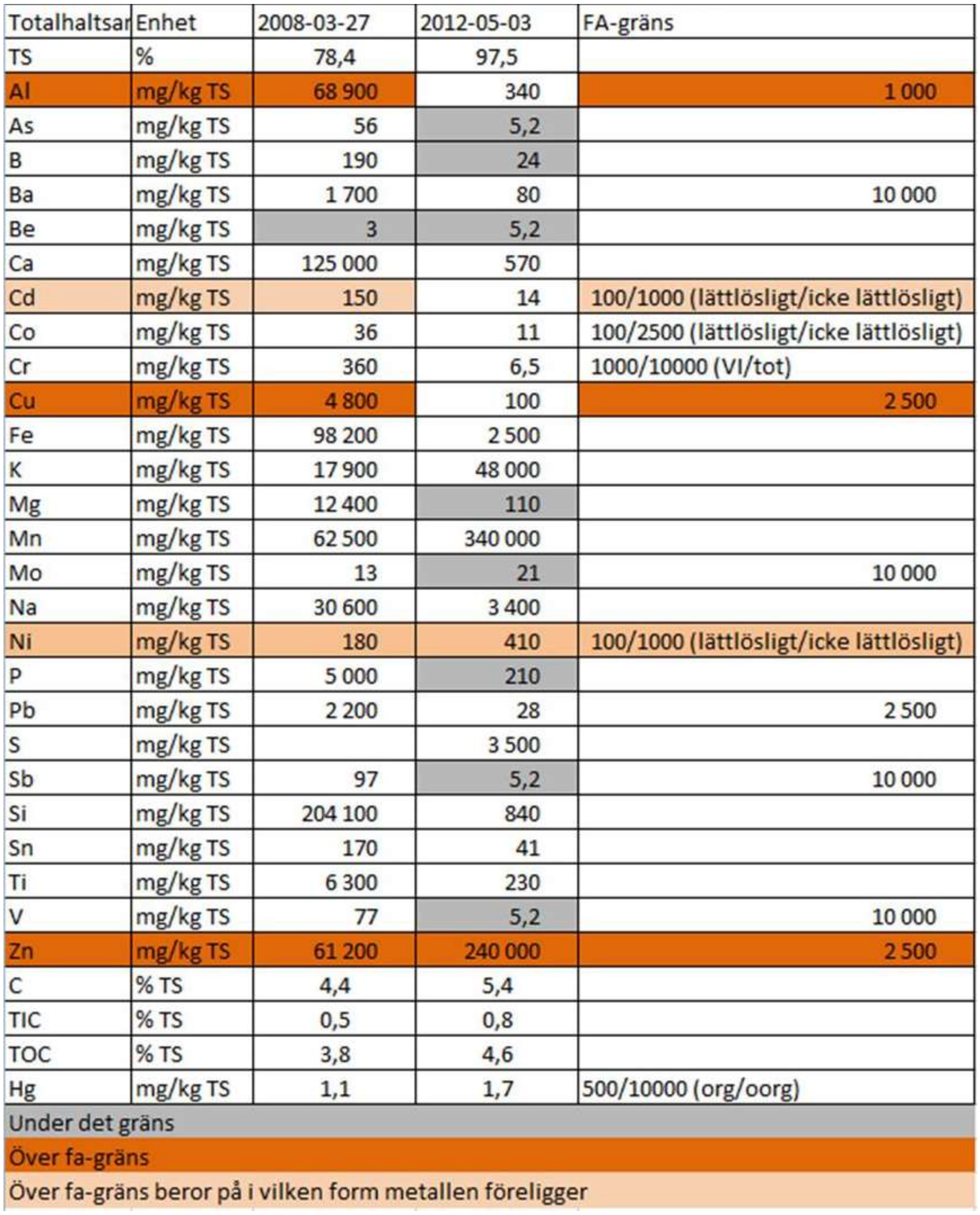
Appendix A: 2012 analysis of 1,5V AA alkaline batteries by Renova



Totalhaltsar	Enhet	2008-03-27	2012-05-03	FA-gräns
TS	%	78,4	97,5	
Al	mg/kg TS	68 900	340	1 000
As	mg/kg TS	56	5,2	
B	mg/kg TS	190	24	
Ba	mg/kg TS	1 700	80	10 000
Be	mg/kg TS	3	5,2	
Ca	mg/kg TS	125 000	570	
Cd	mg/kg TS	150	14	100/1000 (lättlösligt/icke lättlösligt)
Co	mg/kg TS	36	11	100/2500 (lättlösligt/icke lättlösligt)
Cr	mg/kg TS	360	6,5	1000/10000 (VI/tot)
Cu	mg/kg TS	4 800	100	2 500
Fe	mg/kg TS	98 200	2 500	
K	mg/kg TS	17 900	48 000	
Mg	mg/kg TS	12 400	110	
Mn	mg/kg TS	62 500	340 000	
Mo	mg/kg TS	13	21	10 000
Na	mg/kg TS	30 600	3 400	
Ni	mg/kg TS	180	410	100/1000 (lättlösligt/icke lättlösligt)
P	mg/kg TS	5 000	210	
Pb	mg/kg TS	2 200	28	2 500
S	mg/kg TS		3 500	
Sb	mg/kg TS	97	5,2	10 000
Si	mg/kg TS	204 100	840	
Sn	mg/kg TS	170	41	
Ti	mg/kg TS	6 300	230	
V	mg/kg TS	77	5,2	10 000
Zn	mg/kg TS	61 200	240 000	2 500
C	% TS	4,4	5,4	
TIC	% TS	0,5	0,8	
TOC	% TS	3,8	4,6	
Hg	mg/kg TS	1,1	1,7	500/10000 (org/oorg)
Under det gräns				
Över fa-gräns				
Över fa-gräns beror på i vilken form metallen föreligger				

1: 2012 analysis of black mass deriving from 1,5V AA alkaline batteries from Renova

Appendix B: HACH analysis

2: Initial tests to determine which L/S ratio and HCl concentration to exam further, ASSUMPTION: the black mass contains 20% Zn

Sample	[HCl] (M)	L/S	pH	$[Zn]_{HACH}$ (mg/L)	m_{crush}	Zn (mg)	Yield Zn (%)
A	1	1	6.28	0.35	10.05	70	3.5%
B	1	3	0.88	0.51	5.02	153	15.0%
C	1	4	1.10	0.42	5.09	168	16.5%
D	1	5	0.49	0.71	5.00	355	35.5%
E	1	7	0.80	0.69	4.98	483	48.5%
F	0.5	3	4.27	0.84	5.02	252	25.5%
G	1	3	2.07	0.61	5.08	183	18.0%
H	2	3	2.15	0.65	5.06	195	19.5%
I	3	3	1.01	0.73	5.02	219	22.0%

3: HACH analysis results from experiments 1-3

Sample	[Zn] HACH (mg/L)	Dilution factor	$Zn_{supernatant}$ (mg)	Yield Zn (%)
1.1	0.52	20 000	364.0	26.0%
1.2	0.55	22 500	433.1	31.0%
1.3	0.37	25 000	323.8	23.0%
2.1	0.22	20 000	153.0	10.9%
2.2	0.42	20 000	294.0	21.0%
2.3	0.34	20 000	238.0	16.9%
3.1	0.46	20 000	322.0	23.1%
3.2	0.45	20 000	315.0	22.4%
3.3	0.65	20 000	455.0	32.4%

4: HACH analysis when testing accuracy of the method

Sample	V_{ZnCl2} (µL)	V_{Sample} (µL)	Dilution factor	$[Zn]_{HACH}$ (mg/L)	$[Zn]_{calculated}$ (mg/L)
i	2	0	20 000	1.01	0.96
ii	3	0	20 000	1.70	1.44
iii	4	0	20 000	2.10	1.92
iv	0	2	20 000	0.22	-
v	2	2	20 000	0.63	-
vi	3	2	20 000	0.30	-
vii	4	2	20 000	0.40	-

Appendix C: Leaching

5: Results from leaching process. Zn in black mass is based on 2012 analysis of alkaline batteries by Renova

Sample	$m_{\text{black mass}}$ (g)	$m_{\text{blak mass remaining}}$ (g)	Black mass dissolved (%)	pH_{leachate}	c_{Zn} (mg/mL)	c_{Mn} (mg/mL)	Zn recovered (%)
1.1	7.01	5.302	24.37	3.86	18.95	7.35	39.42
1.2	7.00	5.237	25.19	1.58	17.25	7.10	35.94
1.3	7.01	5.468	22.00	2.40	17.40	7.45	36.20
2.1	7.02	4.176	40.51	1.36	37.20	19.05	77.28
2.2	7.00	4.09	41.57	1.48	35.60	17.60	74.17
2.3	7.03	4.179	40.55	0.95	35.55	16.55	73.75
3.1	6.98	5.87	15.90	4.75	8.65	2.56	18.07
3.2	7.02	5.88	16.24	5.03	8.35	2.40	17.35
3.3	7.02	5.96	15.10	4.94	8.55	2.41	17.76
4.1	10.01	8.36	16.48	4.62	17.70	5.05	22.10
4.2	10.02	8.41	16.07	4.50	18.35	5.00	22.89
4.3	9.98	8.38	16.03	5.31	18.05	4.85	22.61
5.1	5.04	3.23	35.91	3.03	19.30	7.80	55.84
5.2	5.02	3.09	38.45	2.84	19.10	7.70	55.49
5.3	5.00	3.13	37.40	2.90	18.75	7.75	54.69
6.1	7.06	5.31	24.79	3.85	19.10	6.35	39.45
6.2	7.05	5.30	24.82	3.95	18.85	6.60	38.99
6.3	7.02	5.31	24.36	3.84	18.55	6.60	38.54
7.1	7.02	4.92	29.91	4.07	19.50	6.55	40.51
7.2	7.02	4.93	29.77	4.00	19.00	6.45	39.47
7.3	6.99	4.90	29.90	4.11	18.9	6.70	39.43
8.1	7.00	3.99	43.00	2.51	36.10	17.55	75.21
8.2	7.02	4.06	42.17	2.48	35.40	17.40	73.54
8.3	7.02	3.80	45.87	2.61	36.35	17.45	75.51
9.1	5.01	2.31	53.89	0.38	32.85	17.95	95.62
9.2	5.02	2.29	54.38	0.41	33.35	18.25	96.88
9.3	5.00	2.10	58.00	0.47	32.50	17.25	94.79

6: The average compositions of Zn, Mn, K, Na, Fe and Cr in the leachate expressed in mg element per kg black mass

Experiment	Zn (mg/kg)	Mn (mg/kg)	K (mg/kg)	Na (mg/kg)	Fe (mg/kg)	Cr (mg/kg)
1	89 248	36 465	35 965.75	1 605.97	115.14	49.95
2	180 154	88 456	36 828.98	1 788.24	568.65	49.88
3	42 543	12 255	37 048.05	1 689.22	49.95	49.95
4	54 082	14 895	36 337.89	1 574.48	29.99	29.99
5	132 819	54 034	38 695.22	1 788.35	69.72	69.72
6	93 587	32 383	37 849.03	1 668.84	49.69	49.69
7	95 530	32 786	38 361.86	1 691.75	49.93	49.93
8	179 408	87 167	38 842.68	1 861.45	49.90	49.90
9	229 840	124 468	60 778.44	2 057.39	2356.62	69.86

7: The average composition of Zn in the leachate and solid residue expressed in mg element per kg black mass. The calculated start value of Zn in the black mass, the percentage of Zn in the selected black mass and average proportion of Zn in the black mass.

Experiment	Zn leachate (mg/kg)	Zn leached black mass (mg/kg)	Zn black mass (mg/kg)	Zn (%)
2	180 154	64 300	244 454	24.45
5	132 819	83 499	216 318	21.63
8	179 408	64 600	244 008	24.40
9	229 840	26 700	256 540	25.65
Average	-	-	-	24.03

Appendix D: Precipitation

8: Results from precipitation process. Zn(OH)2 (%) assumes that all Zn(OH)2 dissolved during analysis

Sample	$pH_{prec.}$	$pH_{prec.\ 30min}$	NaOH (g)	$m_{precipitation}$ (g)	$Zn(OH)_{2\ precip.}$ (%)	Zn precipitated (%)
1.1	7.40	7.24	0.41	0.518	77.04	69.24
1.2	7.25	7.17	0.40	0.522	75.75	75.37
1.3	7.25	7.14	0.38	0.588	70.60	78.45
2.1	7.33	7.08	0.80	1.370	94.53	114.47
2.2	7.26	7.07	0.75	1.255	77.01	89.26
2.3	7.36	7.15	0.79	1.225	79.16	89.69
3.1	7.30	7.32	0.41	0.29	75.57	83.30
3.2	7.27	7.29	0.40	0.27	78.11	83.04
3.3	7.34	7.40	0.38	0.28	97.41	104.89
4.1	7.36	7.33	0.39	0.64	75.72	90.02
4.2	7.33	7.21	0.38	0.63	76.97	86.88
4.3	7.33	7.25	0.37	0.63	76.61	87.92
5.1	7.50	7.19	0.42	0.66	74.45	83.71
5.2	7.70	7.35	0.44	0.66	76.15	86.52
5.3	7.46	7.14	0.41	0.64	82.43	92.51
6.1	7.36	7.26	0.40	0.64	75.29	82.95
6.2	7.34	7.39	0.42	0.74	72.25	93.26
6.3	7.33	7.31	0.42	0.67	74.01	87.89
7.1	7.74	7.26	0.44	0.68	77.91	89.33
7.2	7.36	7.14	0.44	0.64	82.00	90.82
7.3	7.70	7.46	0.43	0.67	72.96	85.04
8.1	7.54	7.07	0.78	1.32	75.88	91.23
8.2	7.32	6.87	0.74	1.27	75.42	88.96
8.3	7.55	7.11	0.79	1.33	72.28	86.96
9.1	7.24	7.31	0.94	1.23	69.38	85.42
9.2	7.70	7.67	0.91	1.22	72.55	87.26
9.3	7.32	7.19	0.82	1.14	81.53	94.03

9: Weight of dissolved precipitation, (%) insoluble precipitation and composition of dissolved precipitation

Sample	**$m_{Precipitation\ dissolved}$**	**Insoluble precipitation (%)**	**Zn (%)**	**$Zn(OH)_2$ (%)**	**Mn (%)**	**Ni (%)**	**Cr (%)**	**Fe (%)**
1.1	0.204	18.14	50.66	77.04	2.05	0.23	0.001	-
1.2	0.203	22.80	49.81	75.75	2.00	0.23	0.003	0.082
1.3	0.203	23.07	46.43	70.60	2.45	0.22	0.001	-
2.1	0.200	24.75	62.16	94.53	4.12	0.29	0.001	0.108
2.2	0.200	27.58	50.64	77.01	2.92	0.22	-	0.140
2.3	0.200	32.55	52.06	79.16	2.76	0.21	0.002	0.326
3.1	0.200	22.66	49.69	75.57	1.10	0.18	-	0.036
3.2	0.201	19.38	51.36	78.11	1.05	0.18	0.001	-
3.3	0.203	20.28	64.06	97.41	1.82	0.24	0.002	0.037
4.1	0.201	18.18	49.79	75.72	1.39	0.22	-	-
4.2	0.201	20.06	50.61	76.97	1.34	0.22	0.001	-
4.3	0.202	21.22	50.38	76.61	1.37	0.22	-	0.013
5.1	0.203	24.04	48.96	74.45	2.69	0.20	0.002	0.000
5.2	0.202	25.54	50.08	76.15	3.39	0.23	0.001	0.094
5.3	0.200	20.27	54.21	82.43	2.35	0.21	0.004	-
6.1	0.200	20.95	49.51	75.29	2.28	0.26	0.002	0.054
6.2	0.200	25.23	47.51	72.25	3.53	0.26	0.003	-
6.3	0.200	19.40	48.66	74.01	3.36	0.25	0.003	0.011
7.1	0.203	18.07	51.23	77.91	2.38	0.30	0.003	-
7.2	0.204	20.25	53.92	82.00	1.77	0.31	-	0.040
7.3	0.200	17.86	47.98	72.96	2.26	0.28	0.002	-
8.1	0.200	27.87	49.90	75.88	4.19	0.22	0.003	0.029
8.2	0.201	23.11	49.60	75.42	3.58	0.23	0.001	0.048
8.3	0.204	19.44	47.53	72.28	4.50	0.22	0.001	0.008
9.1	0.200	19.70	45.63	69.38	4.56	0.22	0.003	0.535
9.2	0.201	19.65	47.70	72.55	6.44	0.23	0.004	0.568
9.3	0.201	22.44	53.61	81.53	3.18	0.26	0.005	0.463

10: Zn and $Zn(OH)_2$ in precipitations

Experiment	Zn (%)	$Zn(OH)_2$ (%)
1.1	50.66	77.04
1.2	49.81	75.75
1.3	46.43	70.60
2.1	62.16	94.53
2.2	50.64	77.01
2.3	52.06	79.16
3.1	49.69	75.57
3.2	51.36	78.11
3.3	64.06	97.41
4.1	49.79	75.72
4.2	50.61	76.97
4.3	50.38	76.61
5.1	48.96	74.45
5.2	50.08	76.15
5.3	54.21	82.43
6.1	49.51	75.29
6.2	47.51	72.25
6.3	48.66	74.01
7.1	51.23	77.91
7.2	53.92	82.00
7.3	47.98	72.96
8.1	49.90	75.88
8.2	49.60	75.42
8.3	47.53	72.28
9.1	45.63	69.38
9.2	47.70	72.55
9.3	53.61	81.53

Appendix E: Full Process

11: The percentage of recovered Zn in the leaching and precipitation step and the degree of recycling for the whole process in each experiment.

Experiment	Zn recovered leaching (%)	Zn precipitated (%)	Degree of recycling (%)
1.1	39.42	69.24	27.30
1.2	35.94	75.37	27.09
1.3	36.20	78.45	28.40
2.1	77.28	114.47	88.46
2.2	74.17	89.26	66.20
2.3	73.75	89.69	66.14
3.1	18.07	83.30	15.05
3.2	17.35	83.04	14.40
3.3	17.76	104.89	18.63
4.1	22.10	90.02	19.90
4.2	22.89	86.88	19.89
4.3	22.61	87.92	19.88
5.1	55.84	83.71	46.75
5.2	55.49	86.52	48.01
5.3	54.69	92.51	50.59
6.1	39.45	82.95	32.73
6.2	38.99	93.26	36.36
6.3	38.54	87.89	33.87
7.1	40.51	89.33	36.19
7.2	39.47	90.82	35.85
7.3	39.43	85.04	33.53
8.1	75.21	91.23	68.61
8.2	73.54	88.96	65.42
8.3	75.51	86.96	65.67
9.1	95.62	85.42	81.68
9.2	96.88	87.26	84.54
9.3	94.79	94.03	89.13

Appendix F: ICP-AES Leachate

12: Composition of leachates expressed in mg element per kg black mass (part 1)

SAMPLE	Ca (mg/kg)	Fe (mg/kg)	K (mg/kg)	Mg (mg/kg)	Na (mg/kg)	Si (mg/kg)	Al (mg/kg)	As (mg/kg)
1.1	<499.3	<49.9	40 192.6	<224.7	1 857.3	146.5	<499.3	<249.6
1.2	<500.0	214.5	35 000.0	<225.0	1 535.0	147.8	<500.0	<250.0
1.3	<499.3	81.1	32 703.3	<224.7	1 425.5	140.8	<499.3	<249.6
2.1	<498.6	418.8	37 891.7	<224.4	1 787.4	254.3	<498.6	<249.3
2.2	<500.0	280	38 000.0	<225.0	1 875.0	236.5	<500.0	<250.0
2.3	<497.9	1005.7	34 601.7	<224.0	1 702.7	253.9	<497.9	<248.9
3.1	<501.4	<50.1	37 106.0	<225.6	1 674.8	111.3	<501.4	<250.7
3.2	<498.6	<49.9	37 393.2	<224.4	1 730.1	103	<498.6	<249.3
3.3	<498.6	<49.9	36 645.3	<224.4	1 662.7	104.5	<498.6	<249.3
4.1	<299.7	<30.0	37 013.0	<134.9	1 618.4	97.7	<299.7	<149.9
4.2	<299.4	<29.9	36 077.8	<134.7	1 541.9	92.5	<299.4	<149.7
4.3	<300.6	<30.1	35 921.8	<135.3	1 563.1	99	<300.6	<150.3
5.1	<694.4	<69.4	39 236.1	<312.5	1 795.1	195.8	<694.4	<347.2
5.2	<697.2	<69.7	38 695.2	<313.7	1 798.8	213.3	<697.2	<348.6
5.3	<700.0	<70.0	38 150.0	<315.0	1 771.0	201.6	<700.0	<350.0
6.1	<495.8	<49.6	37 677.1	<223.1	1 643.4	133.9	<495.8	<247.9
6.2	<496.5	<49.6	38 227.0	<223.4	1 750.0	149.7	<496.5	<248.2
6.3	<498.6	<49.9	37 642.5	<224.4	1 612.9	142.8	<498.6	<249.3
7.1	<498.6	<49.9	38 390.3	<224.4	1 650.3	137.9	<498.6	<249.3
7.2	<498.6	<49.9	37 642.5	<224.4	1 627.8	137.6	<498.6	<249.3
7.3	<500.7	<50.1	39 055.8	<225.3	1 797.6	132.7	<500.7	<250.4
8.1	<500.0	<50.0	38 750.0	<225.0	1 872.5	224.5	<500.0	<250.0
8.2	<498.6	<49.9	38 888.9	<224.4	1 869.7	226.4	<498.6	<249.3
8.3	<498.6	<49.9	38 888.9	<224.4	1 842.2	236.6	<498.6	<249.3
9.1	<698.6	2602.3	40 868.3	<314.4	2 060.9	330.8	<698.6	<349.3
9.2	<697.2	2394.9	41 135.5	<313.7	2 077.7	325.6	<697.2	<348.6
9.3	<700.0	2072	39 550.0	<315.0	2 033.5	328	<700.0	<350.0

13: Composition of leachates expressed in mg element per kg black mass (part 2)

SAMPLE	B (mg/kg)	Ba (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Li (mg/kg)	Mn (mg/kg)
1.1	<49.9	<25.0	68.7	<49.9	<49.9	51.9	<25.0	36 697.6
1.2	<50.0	<25.0	56.3	<50.0	<50.0	194.5	<25.0	35 500.0
1.3	<49.9	<25.0	62.2	<49.9	<49.9	304.6	<25.0	37 196.9
2.1	<49.9	32.9	74.8	67.1	<49.9	319.1	<24.9	94 978.6
2.2	<50.0	<25.0	73.5	62.5	<50.0	262.5	<25.0	88 000.0
2.3	<49.8	<24.9	67.7	63.5	<49.8	258.9	<24.9	82 396.9
3.1	<50.1	<25.1	<50.1	<50.1	<50.1	<25.1	<25.1	12 811.6
3.2	<49.9	<24.9	<49.9	<49.9	<49.9	<24.9	<24.9	11 965.8
3.3	<49.9	<24.9	<49.9	<49.9	<49.9	<24.9	<24.9	11 990.7
4.1	<30.0	<15.0	50	<30.0	<30.0	<15.0	<15.0	15 134.9
4.2	<29.9	<15.0	43	<29.9	<29.9	<15.0	<15.0	14 970.1
4.3	<30.1	<15.0	57.9	<30.1	<30.1	<15.0	<15.0	14 579.2
5.1	<69.4	<34.7	79.5	<69.4	<69.4	86.5	<34.7	54 166.7
5.2	<69.7	<34.9	84	<69.7	<69.7	69	<34.9	53 685.3
5.3	<70.0	<35.0	79.5	<70.0	<70.0	83.3	<35.0	54 250.0
6.1	<49.6	<24.8	76.8	<49.6	<49.6	35.7	<24.8	31 480.2
6.2	<49.6	<24.8	65.5	<49.6	<49.6	32.8	<24.8	32 766.0
6.3	<49.9	<24.9	62.1	<49.9	<49.9	33.2	<24.9	32 906.0
7.1	<49.9	<24.9	<49.9	51.9	<49.9	<24.9	<24.9	32 656.7
7.2	<49.9	<24.9	64.1	<49.9	<49.9	39.6	<24.9	32 158.1
7.3	<50.1	<25.0	63.6	<50.1	<50.1	40.1	<25.0	33 547.9
8.1	<50.0	32.3	81.5	67.8	<50.0	125.5	<25.0	87 750.0
8.2	<49.9	33.7	80	68.8	<49.9	122.2	<24.9	86 752.1
8.3	<49.9	33.2	89.5	66.8	<49.9	117.2	<24.9	87 001.4
9.1	<69.9	76.5	102	85.9	<69.9	348.3	38.1	125 399.2
9.2	<69.7	71.1	95.2	81.2	<69.7	355.6	36.3	127 241.0
9.3	<70.0	83.3	112.4	84	<70.0	378	<35.0	120 750.0

14: Composition of leachates expressed in mg element per kg black mass (part 3)

SAMPLE	Mo (mg/kg)	Ni (mg/kg)	P (mg/kg)	Pb (mg/kg)	Sr (mg/kg)	V (mg/kg)	Zn (mg/kg)
1.1	<49.9	474.3	<499.3	<249.6	<25.0	<25.0	**94 614.8**
1.2	<50.0	427.5	<500.0	<250.0	<25.0	<25.0	**86 250.0**
1.3	<49.9	454.4	<499.3	<249.6	<25.0	<25.0	**86 875.9**
2.1	<49.9	852.6	<498.6	<249.3	<24.9	<24.9	**185 470.1**
2.2	<50.0	780	<500.0	<250.0	<25.0	<25.0	**178 000.0**
2.3	<49.8	746.8	<497.9	<248.9	<24.9	<24.9	**176 991.5**
3.1	<50.1	200.1	<501.4	<250.7	<25.1	<25.1	**43 373.9**
3.2	<49.9	195.7	<498.6	<249.3	<24.9	<24.9	**41 631.1**
3.3	<49.9	183.5	<498.6	<249.3	<24.9	<24.9	**42 628.2**
4.1	<30.0	265.2	<299.7	<149.9	<15.0	<15.0	**53 047.0**
4.2	<29.9	253	<299.4	<149.7	<15.0	<15.0	**54 940.1**
4.3	<30.1	255.5	<300.6	<150.3	<15.0	<15.0	**54 258.5**
5.1	<69.4	583.3	<694.4	<347.2	<34.7	<34.7	**134 027.8**
5.2	<69.7	606.6	<697.2	<348.6	<34.9	<34.9	**133 167.3**
5.3	<70.0	574	<700.0	<350.0	<35.0	<35.0	**131 250.0**
6.1	<49.6	525.5	<495.8	<247.9	<24.8	<24.8	**94 688.4**
6.2	<49.6	548.6	<496.5	<248.2	<24.8	<24.8	**93 581.6**
6.3	<49.9	513.5	<498.6	<249.3	<24.9	<24.9	**92 485.8**
7.1	<49.9	588.3	<498.6	<249.3	<24.9	<24.9	**97 222.2**
7.2	<49.9	580.8	<498.6	<249.3	<24.9	<24.9	**94 729.3**
7.3	<50.1	608.4	<500.7	<250.4	<25.0	<25.0	**94 635.2**
8.1	<50.0	850	<500.0	<250.0	<25.0	<25.0	**180 500.0**
8.2	<49.9	807.7	<498.6	<249.3	<24.9	<24.9	**176 495.7**
8.3	<49.9	835.1	<498.6	<249.3	<24.9	<24.9	**181 232.2**
9.1	<69.9	1107.3	<698.6	<349.3	<34.9	<34.9	**229 491.0**
9.2	<69.7	1146.9	<697.2	<348.6	<34.9	<34.9	**232 519.9**
9.3	<70.0	1120	<700.0	<350.0	<35.0	<35.0	**227 500.0**

Appendix G: ICP-MS Precipitations

15: Compositions of precipitation expressed in mg element per kilo precipitation (part 1)

SAMPLE	Li (mg/kg)	Ti (mg/kg)	V (mg/kg)	Cr (mg/kg)	Mn (mg/kg)	Fe (mg/kg)
1.1	6.1	0.0	0.0	9.0	20 477.5	0.0
1.2	8.6	0.0	0.0	34.8	20 046.5	816.5
1.3	9.1	0.0	0.0	10.9	24 472.7	0.0
2.1	7.3	0.0	0.0	8.4	41 163.3	1084.2
2.2	5.3	0.0	0.0	2.1	29 227.2	1400.7
2.3	5.0	0.0	0.0	20.9	27 589.3	3262.3
3.1	4.9	0.0	0.0	0.0	11 025.1	359.1
3.2	4.9	0.0	0.0	6.7	10 515.6	0.0
3.3	4.0	0.0	0.0	21.1	18 210.7	372.8
4.1	5.4	0.0	0.0	0.0	13 864.2	0.0
4.2	5.2	0.0	0.0	11.2	13 382.3	0.0
4.3	4.8	0.0	0.0	3.9	13 706.4	125.9
5.1	4.8	0.0	0.0	17.2	26 871.9	0.0
5.2	3.9	0.0	0.0	5.7	33 871.4	936.3
5.3	3.9	263.3	0.0	37.9	23 496.1	0.0
6.1	4.9	0.0	0.0	17.0	22 791.2	535.9
6.2	5.5	209.7	0.0	33.8	35 300.9	0.0
6.3	5.7	0.0	0.0	28.2	33 596.9	114.0
7.1	5.2	0.0	0.4	26.8	23 764.1	0.0
7.2	3.0	0.0	0.0	3.6	17 748.6	401.6
7.3	4.2	0.0	1.1	22.3	22 590.2	0.0
8.1	4.0	0.0	0.0	26.5	41 903.7	290.8
8.2	3.4	0.0	0.0	7.4	35 811.1	476.1
8.3	4.2	0.0	0.0	10.0	45 044.8	80.2
9.1	5.2	0.0	7.6	32.1	45 563.0	5354.5
9.2	4.1	0.0	0.0	35.5	64 412.0	5678.8
9.3	6.1	173.1	0.0	45.9	31 787.0	4630.6

16: Compositions of precipitation expressed in mg element per kilo precipitation (part 2)

SAMPLE	Ni (mg/kg)	Cu (mg/kg)	Zn (mg/kg)	Cd (mg/kg)	Pb (mg/kg)
1.1	2266.3	204.3	**506 588.3**	40.2	3.4
1.2	2254.0	989.2	**498 122.6**	50.6	7.5
1.3	2168.5	1412.8	**464 274.3**	49.4	0.0
2.1	2887.6	1075.7	**621 622.2**	39.2	0.0
2.2	2182.5	651.2	**506 398.1**	23.9	0.0
2.3	2127.7	666.9	**520 556.0**	16.9	15.6
3.1	1773.9	47.5	**496 913.3**	53.4	0.0
3.2	1814.4	48.5	**513 610.8**	50.5	0.0
3.3	2429.7	99.6	**640 577.8**	61.5	0.0
4.1	2161.5	5.3	**497 900.6**	55.5	0.0
4.2	2192.4	19.0	**506 122.9**	40.6	0.0
4.3	2210.8	7.7	**503 789.9**	57.4	0.0
5.1	2001.4	248.3	**489 597.1**	30.1	0.0
5.2	2282.5	166.2	**500 771.9**	47.8	0.0
5.3	2078.3	246.0	**542 063.3**	40.3	2.4
6.1	2610.4	93.9	**495 087.6**	53.4	0.0
6.2	2643.8	92.5	**475 131.3**	74.3	0.0
6.3	2472.7	93.2	**486 649.7**	53.0	0.2
7.1	3013.2	70.5	**512 345.6**	32.9	0.0
7.2	3113.1	126.5	**539 223.7**	28.8	1.7
7.3	2810.5	141.9	**479 799.3**	46.0	0.0
8.1	2177.1	252.8	**499 005.9**	36.8	0.0
8.2	2277.2	265.2	**495 952.1**	33.3	0.0
8.3	2193.0	214.1	**475 332.6**	39.1	0.7
9.1	2219.0	587.8	**456 265.1**	38.9	0.6
9.2	2303.7	628.4	**477 047.8**	57.9	0.0
9.3	2584.3	793.8	**536 134.0**	35.6	10.2

Appendix H: ICP-AES Filtrate

17: Composition of leachates expressed in mg element per kg black mass (part 1)

SAMPLE	Ca (mg/L)	Fe (mg/L)	K (mg/L)	Mg (mg/L)	Na (mg/L)	Si (mg/L)	Al (mg/L)	As (mg/L)
2.1	<100	<10	4 855	<45	15 900	<20	<100	<50
2.2	<100	<10	4 810	<45	14 650	<20	<100	<50
2.3	<100	<10	4 190	<45	15 100	<20	<100	<50
5.1	<100	<10	4 145	<45	9 500	<20	<100	<50
5.2	<100	<10	3 755	<45	8 950	<20	<100	<50
5.3	<100	<10	3 610	<45	8 100	<20	<100	50
8.1	<100	<10	4 750	<45	14 400	<20	<100	<50
8.2	<100	<10	4 820	<45	14 200	<20	<100	<50
8.3	<100	<10	4 520	<45	14 200	<20	<100	<50
9.1	<100	<10	3 135	<45	14 350	<20	<100	<50
9.2	<100	<10	3 120	<45	15 050	<20	<100	<50
9.3	<100	<10	3 095	<45	13 850	<20	<100	<50

18: Composition of leachates expressed in mg element per kg black mass (part 2)

SAMPLE	B (mg/L)	Ba (mg/L)	Cd (mg/L)	Co (mg/L)	Cr (mg/L)	Cu (mg/L)	Li (mg/L)	Mn (mg/L)
2.1	<10	6.25	<10	<10	<10	<5	<5	10 750
2.2	<10	5.55	<10	<10	<10	<5	<5	10 050
2.3	<10	7.6	<10	<10	<10	<5	<5	8 800
5.1	<10	<5	<10	<10	<10	<5	<5	5 200
5.2	<10	<5	<10	<10	<10	<5	<5	4 455
5.3	<10	<5	<10	<10	<10	<5	<5	4 835
8.1	<10	<5	<10	<10	<10	<5	<5	9 250
8.2	<10	<5	<10	<10	<10	<5	<5	9 600
8.3	<10	<5	<10	<10	<10	<5	<5	8 400
9.1	<10	6.3	<10	<10	<10	<5	<5	7 850
9.2	<10	6	<10	<10	<10	<5	<5	6 450
9.3	<10	6.65	<10	<10	<10	<5	<5	8 450

19: Composition of leachates expressed in mg element per kg black mass (part 3)

SAMPLE	Mo (mg/L)	Ni (mg/L)	P (mg/L)	Pb (mg/L)	Sr (mg/L)	V (mg/L)	Zn (mg/L)
2.1	<10	<20	<50	<50	<5	<5	**188**
2.2	<10	<20	<50	<50	<5	<5	**209.5**
2.3	<10	<20	<50	<50	<5	<5	**156.5**
5.1	<10	<20	<50	<50	<5	<5	**125**
5.2	<10	<20	<50	<50	<5	<5	**78**
5.3	<10	<20	<50	<50	<5	<5	**121**
8.1	<10	<20	<50	<50	<5	<5	**96.5**
8.2	<10	<20	<50	<50	<5	<5	**191.5**
8.3	<10	<20	<50	<50	<5	<5	**95**
9.1	<10	<20	<50	<50	<5	<5	**85**
9.2	<10	<20	<50	<50	<5	<5	**20.8**
9.3	<10	<20	<50	<50	<5	<5	**106**

Appendix I: ICP-SFMS Solids

20: Compositions of solids expressed in mg element per kg black mass (analysis from 2017)

ELEMENT	ROUGH MASS (MG/KG)	SORTED MASS (MG/KG)	EXPERIMENT 2 SOLID RESIDUE (MG/KG)	EXPERIMENT 5 SOLID RESIDUE (MG/KG)	EXPERIMENT 8 SOLID RESIDUE (MG/KG)	EXPERIMENT 9 SOLID RESIDUE (MG/KG)
Si	<1000	<1000	<1000	<1000	<1000	<1000
Al	341	378	1 690	440	492	547
Ca	<500	<500	<500	<500	<500	<500
Fe	3 340	4 090	4 890	5 200	9 250	20 600
K	26 300	26 300	5 050	2 990	3 670	4 170
Mg	91	106	80	80	79	75
Mn	176 000	182 000	186 000	209 000	242 000	287 000
Na	<1000	1090	<1000	<1000	<1000	<1000
P	<50	<50	<50	<50	<50	<50
Ti	274	316	341	290	413	458
As	<5	<5	<5	<5	<5	<5
Ba	835	661	912	1 010	1 240	1 510
Be	<1	<1	<1	<1	<1	<1
Cd	59.4	69.4	17	22.1	13.9	7.48
Co	53.5	62.5	456	60.3	56.4	54
Cr	<5	<5	10.5	5.3	6.7	8.8
Cu	220	251	223	450	351	204
Hg	7.34	10.2	4.38	12.9	9.27	6.34
Nb	<0.5	<0.5	<0.5	<0.5	0.777	<0.5
Ni	700	888	391	835	671	633
Pb	73.7	76.6	262	103	146	187
S	1070	1120	820	<500	720	1270
Sc	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Sr	18.1	20	15.3	15.8	18.8	23.9
V	1.58	1.6	2.42	1.71	2.68	3.94
W	0.479	0.475	8.24	0.657	0.874	1.28
Y	1.62	1.93	1.14	1.62	1.47	1.17
Zn	**127 000**	**131 000**	**64 300**	**83 400**	**64 600**	**26 700**
Zr	<1	<1	1.5	<1	3.04	3.12

Appendix J: Calculations

Leaching

$$m_{Zn,black\ mass} = m_{black\ mass} * Zn_{black\ mass} \qquad \text{(g)}$$

$$Dissolved\ black\ mass = \frac{m_{dissolved\ black\ mass}}{m_{black\ mass}} = 1 - \frac{m_{black\ mass\ after\ leaching}}{m_{\ black\ mass}} \qquad \text{(\%)}$$

$$c_{Zn\ leachant} = c_{Zn\ leachant\ analysis} * dilution\ factor = c_{Zn\ leachant\ analysis} * 5 \qquad \text{(mg/L)}$$

$$m_{Zn\ leachant} = c_{Zn\ leachant} * V_{leaching\ media} * 10^{-3} \qquad \text{(mg)}$$

$$Zn_{recovered\ leachin} = \frac{m_{Zn\ leachant}}{m_{Zn\ black\ mass}*1000} \qquad \text{(\%)}$$

All concentrations of different elements in the leachate were calculated according to:

$$c_{element,leachant} = c_{element\ leachant\ analysis} * 5 \qquad \text{(mg/L)}$$

The leachate concentrations in the ICP-MS analysis are expressed as mg element in leachate per kg black mass according to:

$$\frac{m_{element}}{m_{black\ mass}*10^{-3}} = \frac{c_{element\ leachant\ analysis}*5*V_{leaching\ media}}{m_{black\ mass}*10^{-3}} \qquad \text{(mg/kg)}$$

The average Zn composition of black mass from experiment 2, 5, 8 and 9 were calculated according to:

$$c_{Zn,black\ mass} = c_{Zn\ leached\ black\ mass} + c_{Zn\ leachant} \qquad \text{(mg/kg)}$$

$$Zn_{black\ mass} = \frac{c_{Zn,black\ mass}}{10\ 000} \qquad \text{(\%)}$$

Precipitation

$$insoluble\ precipitation = \frac{m_{insolubl\ \ precipitation}}{m_{precipitation\ added}} \qquad \text{(\%)}$$

$$c_{Zn\ solution,5mL} = \frac{c_{Zn\ ICP}*dillution\ factor}{1\ 000} = \frac{c_{Zn\ ICP}*25\ 000}{1\ 000} \qquad \text{(mg/L)}$$

$$c_{precipitation\ solution,5mL} = \frac{m_{added\ precip}*1000}{5*10^{-3}} \qquad \text{(mg/L)}$$

$$c_{Zn\ precip} = \frac{c_{Zn,\ \ solution,5mL}}{Precip\ cons_{solution,5mL}} \qquad \text{(\%)}$$

$$Zn_{precip} = \frac{C_{Zn\ precip}}{c_{precipitation\ solution,5mL}} \qquad \text{(\%)}$$

$$Zn(OH)_{2_{precip}} = \frac{m_{Zn(OH)_{2_{precip}}}}{m_{added\ precip}} = \frac{n_{Zn(OH)_{2_{precip}}}*M_{Zn(OH)_2}}{m_{added\ precip}} =$$

$$\frac{m_{added\ precip}*Zn_{precip}*M_{Zn(OH)_2}}{M_{Zn}*m_{added\ precip}} = \frac{Zn_{precip}*M_{Zn(OH)_2}}{M_{Zn}} \qquad \text{(\%)}$$

$$m_{Zn_{precip}} = m_{added\ precip} * Zn_{precip} \qquad \text{(mg)}$$

$$m_{Zn_{leachant\ 20mL}} = c_{Zn\ leachant} * 20mL \qquad \text{(mg)}$$

$$Zn_{recovered\ precipitation} = \frac{m_{Zn_{precip}}}{m_{Zn_{leachant}}} \qquad \text{(\%)}$$

All concentrations of different elements in the i) precipitations and ii) filtrates were calculated according to:

$$i)\ c_{element,precipitation} = \frac{c_{element\ analysis\ precipitation}}{1\ 000} * 25\ 000 \qquad \text{(mg/L)}$$

$$ii)\ c_{element,filtrate} = c_{element\ analysis\ filtrate} * dillution\ factor = c_{element\ \ analysis\ filtrate} * 5 \quad \text{(mg/L)}$$

For the ICP-AES analysis of the precipitations, the concentrations in the precipitations, expressed in mg element in precipitation per kg precipitation, were calculated according to:

$$\frac{1000 * m_{element}}{m_{added\ precip} * 10^{-3}} = \frac{c_{element\ analysis\ precipitation} * 25000 * V_{HNO3,\ \ 5\ mL,}}{1000 * m_{\ added\ precip} * 10^{-3}} \qquad \text{(mg/kg)}$$

As for the ICP-AES analysis of the filtrate, the concentrations are expressed as mg element per L filtrate, i.e $c_{\text{element, filtrate}}$ calculated according to ii).

Full process

$$Degree\ of\ recycling = Zn_{recovered\ leaching} * Zn_{precipitated} \qquad \text{(%)}$$

Designations

$m_{\text{Zn black mass}}$: Mass Zn in the samples (g)
m_{element}: Mass element in the samples (mg)
$m_{\text{black mass}}$: Weight of black mass in each sample (g)
$Zn_{\text{black mass}}$: Percent Zn in black mass (%)
Dissolved black mass: Percent of black mass that dissolved during leaching (%)
$m_{\text{dissolved black mass}}$: Weight of dissolved black mass (g)
$m_{\text{black mass after leaching}}$: Weight of black mass remaining after leaching (g)
$c_{\text{Zn leachate}}$: Concentration Zn in leachate (mg/L)
$c_{\text{Zn leachate analysis:}}$ Concentration Zn in the ICP analysis (mg/L)
Dilution factor: The dilution factor of the original samples before analysis
$m_{\text{Zn leachate}}$: Mass Zn in leachate (g)
$V_{\text{leaching media}}$: Volume of HCl used in leaching process (mL)
V_{Hno3}: Volym of HNO_3 used to dissolve precipitation (L)
$Zn_{\text{recovered leaching}}$: Percent Zn recovered from black mass during leaching (%)
$c_{\text{element, leachate}}$: Concentration of an element present in leachate (mg/L)
Insoluble precipitation: Percent of precipitation that did not dissolve in HNO_3 (%)
$m_{\text{insoluble precipitation}}$: Weight of insoluble precipitation (g)
$m_{\text{added precip}}$: Mass precipitation added to 5mL solution (g)
$c_{\text{Znsolution, 5mL}}$: Concentration of Zn in the 5mL solution 0,2 precipitation dissolved in (mg/L)
c_{ZnICP}: Concentration of Zn in the dissolved precipitation analyzed by ICP-MS (ppb)
$c_{\text{elementICP}}$: Concentration of element in the dissolved precipitation analyzed by ICP-MS (ppb)
$c_{\text{Precipitation solution, 5mL}}$: Concentration precipitation in 5mL solution (mg/L)
Zn_{precip}: Percent Zn in precipitation (%)
$Zn(OH)_{2\ \text{precip}}$: Percent $Zn(OH)_2$ in precipitation (%)
$m_{\text{zn, precip}}$: Mass Zn in precipitation (mg)
$m_{\text{zn, leachate}}$: Mass Zn in leachate (mg)
$Zn_{\text{recovered precipitation}}$: Percent Zn precipitated from Zn present in leachate (%)
$c_{\text{element precipitation}}$: Concentration of an element present in the ICP analysis of the precipitation (mg/L)

$c_{elementICP}$: Concentration of an element present in the ICP analysis of the filtrate	(mg/L)
$c_{element}$: Concentration of an element present in the filtrate	(mg/L)
Degree of recycling: Percent recycled Zn from black mass	(%)
Zn_{precip}: Percent Zn in precipitation	(%)
$Zn(OH)_{2\ precip}$: Percent $Zn(OH)_2$ in precipitation	(%)
$m_{zn,\ precip}$: Mass Zn in precipitation	(mg)
$m_{zn,\ leachate}$: Mass Zn in leachate	(mg)
$Zn_{recovered\ precipitation}$: Yield of precipitation process	(%)
$c_{element\ precipitation}$: Concentration of an element present in the ICP analysis of the precipitation	(mg/mL)
$c_{elementICP}$: Concentration of an element present in the ICP analysis of the filtrate	(mg/L)
$c_{element}$: Concentration of an element present in the filtrate	(mg/L)
Degree of recycling: Percent recycled Zn from black mass	(%)