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Leaching and Recovery of Stainless Steel Production Dusts in Acidic Media

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Abstract

In this paper hydrometallurgical treatment of two different stainless steel plant production dusts (EAF – electric arc furnace and AOD – argon oxygen decarburization converter dusts) are studied. The main objective at the first stage is to screen parameters to i) maximize the zinc dissolution and to ii) test parameters for the best zinc selectivity into solution vs. Cr, Ni, Fe and Mo. The purpose is to recycle these metals back to stainless steel production as solids without harmful Zn. The effect of sulphuric acid concentration, temperature and liquid to solid ratio is tested. Leaching tests are done in 0.1 M, 0.5 M and 1.5 M sulphuric acid and in temperatures 30 °C, 60 °C and 90 °C with liquid to solid ratios of L:S = 10 and 20 in atmospheric pressure.

For EAF dust, maximum Zn dissolution of 60 – 70 % was achieved with 1.5 M – 90 °C – L:S = 10 and 20 and 1.5 M – 60 °C – L:S = 10 parameter groups after 120 min. At the same time however, around 8 % of Cr, 25 – 50 % of Ni and 40 – 60 % of Fe was extracted and Mo almost totally extracted into the solution phase. The highest selectivity for Zn from EAF dust was achieved with 0.5 M – L:S = 10 in temperatures T = 30 °C and 90 °C after 120 min. However, only around 33 – 36 % of Zn and at the same time 0 – 10 % of Cr, Fe, Ni and Mo were extracted. In the AOD dust case, the maximum Zn extraction of 95 % were achieved with 1.5 M – 60 °C and 90 °C – L:S = 10 and 0.5 M – 90 °C – L:S = 20 after 120 min. With the same parameters, Cr was extracted about 10 %, Ni 25 – 45 %, Fe 35 – 55 % and Mo 65 – 85 % into the solution phase. The highest zinc selectivity was achieved already after 20 min with 0.5 M – L:S = 10 in T = 30 °C and 90 °C. Around 80 % of Zn was extracted and at the same time Cr was extracted around 5 %, Ni around 10 %, Fe 8 – 15 % and Mo 20 – 60 %. AOD dust has better hydrometallurgical recycling possibilities than EAF dust due to better Zn yields and selectivity into solution. This is probably due to differences in

chemical and mineralogical properties, i. e. alkaline Ca and zinc – ferrite phases are found to be more in this batch of EAF dust than in AOD dust.

1 Introduction

In stainless steel production various amounts of valuable metal containing dusts are generated in different parts of the process. For example in electric arc furnace (EAF) about 1 – 2 % of the charge is turned into different elements containing dust [1, 2]. EAF dust formation is estimated to be 6.4 million tons per year worldwide and in Europe 1.3 million tons/a [3]. The EAF and AOD dusts contains various amounts of chromium, nickel, iron, zinc and other heavy metal oxides that are classified harmful to environment and the content of these dusts may vary from day to day [2, 4, 5]. Due to tightening environmental legislation and high price of alloying compounds, the valuable part (Cr, Ni, Mo) of the stainless steel dusts are in the interest to recycle back to the process, and the rest, partly harmful materials that are land filled to be minimized or neutralized. Today, carbon steel and stainless steel dusts are treated in separate plants mainly by pyrometallurgical methods to recover part of the valuable materials (Zn, Cr, Ni, Mo, Fe) for recycling. However, a large part is still stored as landfill. Waelz kiln is the most used recycling technique, where the dust is carbothermically reduced and zinc evaporated and oxidized again [1, 3, 6].

Many hydrometallurgical dust treatment processes have been tested in the past three decades but virtually none of them have passed to wider industrial scale use. The main problem in the acidic dust leaching is low yield and separability of zinc because usually over 50 % of Zn in the dusts exist in zinc ferrite structure $ZnFe_2O_4$ (franklinite) which makes the selective leaching of Zn without Fe difficult, whereas ZnO (zincite) phase does not cause problems in leaching [7 - 10]. Depending on its current price, recovery method and amount in the dust composition, zinc is a valuable material for recycling to primary zinc production but a major impurity if recycled back to stainless steel making process furnaces. Zinc is a volatile component which recirculates and adds up in the furnace. [5]

In the research field of hydrometallurgical treatment methods for steel production dusts, Zn - Fe separation and zinc recovery from carbon steel making dusts, mainly from EAF dusts, has been the most researched topic. Only very few research has been focused on other types of dusts, i.e. AOD dust, or in general, on stainless steel production dusts to recover Cr, Ni, Mo and Fe. So a developed method to recover these valuable elements by hydrometallurgical means is unknown. This might be due to the fact that stainless steel dust recycling by pyrometallurgical ways is quite well established and the value of the recovered elements has been sufficiently high to offset the high-energy consumption, generation of worthless residues to landfill and high CO_2 production [2, 5].

EAF carbon steel dust leaching and zinc recovery routes have been widely studied with different acids, mainly by sulphuric acid and hydrochloric acid, both with atmospheric and elevated pressures. One of the best selectivity yields for zinc with sulphuric acid have been reported to be 84 %

with diluted sulphuric acid of 0.4 mol/dm³ in high pressure of 4.1 bar and temperature 150 °C. At this environment the Fe yield into solution was only 2 % because the use of low concentration of the acid [11]. Another study reported zinc yield to be as high as 92 % using 0.3 M sulphuric acid solution at 260 °C in elevated pressure with microwave heating after 100 min. Fe (II) remained in the solution while most of the Fe (III) precipitated as hematite. In the presence of hydrogen peroxide the zinc yield reached as high as 99 % while only 3 % of the iron remained in solution (11 % without peroxide) with the same parameters. The elevated pressure was only due to the vapor pressure of the acid solution [8]. In atmospheric pressure, maximum yield of almost 100 % Zn into solution was achieved with 3 M H_2SO_4 at 80 °C after 6 h, however 90 % of the Fe was also extracted.

Good selectivity for Zn in the same study was achieved with 0.1-0.3M H_2SO_4 at 80 °C. However, in that range zinc extraction was only 30 % with Zn/Fe ratio about 9. The leach liquor was treated using goethite precipitation [12]. There are also numerous other reports of EAF carbon steel dust leaching and treatment experiments at atmospheric pressure with sulphuric acid but the results vary greatly depending on the dust composition and leaching conditions [2, 5, 7, 13-15].

Hydrochloric acid is found to be a quite effective lixiviant for the steel dusts zinc ferrite spinel and it is reported it can be dissolved in 2 M HCl [16]. Selective leaching of zinc from zinc ferrite have been studied also in diluted HCl 0.3 M [17]. More than 90 % of zinc can be extracted in 250 – 260 °C after 100 min while over 98 % of iron remained in the solid residue. The solid residue from the used TF-sludge contained 98 % Fe_2O_3 and < 0.2 % Zn allowing it to be directly used as a recycling material in iron smelting or used as a pigment. The leach liquor contained less than 3 g/l of iron which can be removed by precipitation after Fe (II) oxidation [17].

M. Jha et al. have done a review of different hydrometallurgical dust treatment processes for zinc recovery from industrial wastes in 2001 [18]. T. Havlik et al. [11], R. Nyirenda et al. [5] and A.D. Zunkel [19] discuss generally hydrometallurgical methods and problems encountered in carbon steel (EAF) dust treatment. For example, it is known fact that Cl and F are detrimental elements with very small amounts in the liquid phase when considering recycling zinc back to primary metallurgy from various steel dusts. The same problems and methods apply to some extent also in stainless steel dust treatment, i. e. leaching harmful Zn as an impurity from the recyclable solids (Cr, Ni, Mo, Fe) and waste liquid management.

The main focus of the present work is on two stainless steel production dusts (EAF and AOD) and the purification of chromium, nickel, iron and molybdenum out of zinc with sulphuric acid. The main objective is to i) maximize zinc extraction into solution and ii) selectively leach maximum amount of zinc leaving the valuable elements Cr, Ni, Fe, Mo in the solid residue for further treatment and possibly recycling back to stainless steel process.

2 Experimental

Two dust materials, from EAF and AOD converter, were received from Outokumpu Stainless (Tornio) for the leaching experiments. The chemical compositions of the stainless steel production dusts are presented in Table 1. The chemical analysis was made by melting and dissolution of the dust and analysed with ICP-AES (Labtium Oy). More detailed characterization and mineralogical analysis of the production dusts is available in another EMC 2011 publication: "Chemical and structural characterization of steelmaking dust from stainless steel production" (F. Kukurugya, T. Havlik, H. Makkonen, 2010).

Table 1: Chemical composition of received dust materials from stainless steel (SS) production

Sample	wt.-%	Zn	Fe	Cr	Ni	Mo	Mn	Ca	Pb
EAF2 SS		5.9	20	15	2.6	0.1	3.1	13.1	1
AOD1 SS		10	33	9.7	0.7	0.05	2.8	4.8	0.1

Typically the composition of one dust may vary from day to day and is dependent on the feed material. In carbon steel production dusts, i.e. EAF dust, the zinc amount is typically 15 – 35 wt.-%, or even up to 40 %, which is generally much higher than in dusts from stainless steel production [9, 11].

Leaching experiments were performed in a 1000 ml glass reactor in a temperature controlled water bath with a cap that had sealable holes for dust material feed, thermometer, stirrer (300 rpm) and cooling colon for water evaporation. Total volume of 600 ml 0.1 M, 0.5 M and 1.5 M sulphuric acid was used for leaching the dust. The dust sample weight was 60 g and 30 g for liquid to solid ratios of 10 and 20, respectively. The temperature range was 30 °C, 60 °C and 90 °C and the liquid samples for chemical analysis were taken after 10, 20, 60 and 120 min.

The samples (10 ml) were filtered and sealed before sending them to multi-element ICP-AES analysis.

3 Results and Discussion

3.1 EAF dust

3.1.1 Effect of temperature and acid concentration

With higher temperatures the dissolution rate for both Zn and Fe is as expected faster. Figure 1 shows the dependence of temperature on zinc and iron dissolution in 0.5 M acid and L:S = 20.
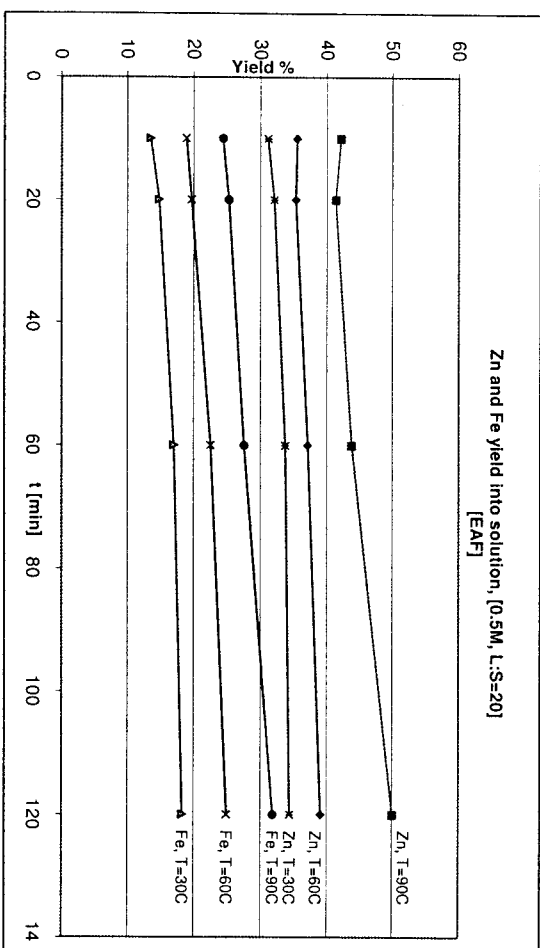
Figure 1: The effect of temperature on zinc and iron dissolution into solution for EAF dust, 0.5 M acid, L:S = 20

However, in dilute 0.1 M acid and L:S = 20, zinc dissolution is quite fast at first but, as the pH increases during the experiment due to the alkaline dust, Zn precipitates back to residue. Fe is not leached at all as pH is too high and the acid is used very fast in neutralizing the alkaline dust (Figure 2).
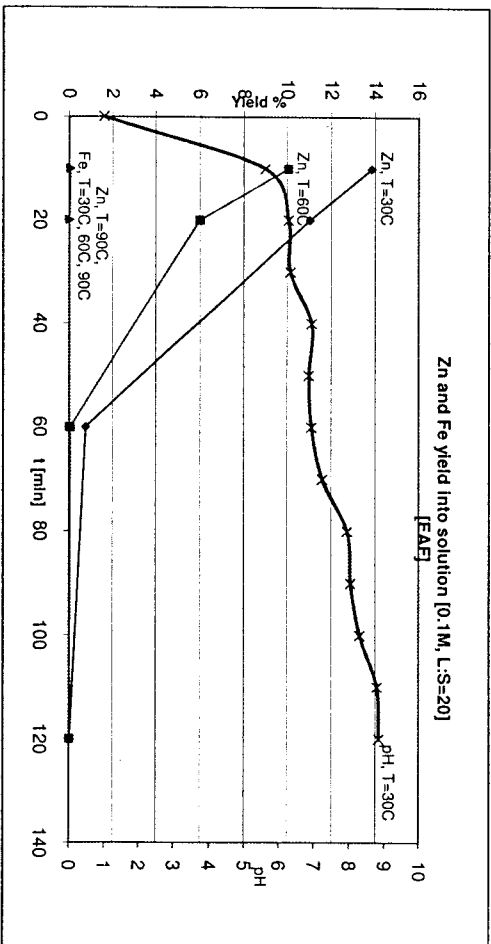
Figure 2: Zinc and iron dissolution in 0.1 M, L:S = 20

The EAF SS dust itself is very alkaline due to high concentration of Ca. Therefore, enough acid is needed to adjust the pH to area of possible Zn dissolution and preferably to area in which iron is left in residue as i. e. FeO-OH or as hematite Fe_2O_3, which can be recycled back to steelmaking furnaces, as iron hydroxides cannot. Hematite precipitation, however, needs high temperatures and possibly high pressure leaching.

With the same 0.1 M acid concentration but with more dust (L:S = 10), zinc is not leached at all as average pH increases rapidly to 12.5 (not in figure).

The effect of acid concentration on zinc and iron yield is shown in Figure 3. As expected, more concentrated acid 0.1 M → 1.5 M enhances the extraction kinetics for both Zn and Fe. However for extraction to continue, enough free acid must be available. This means that pH should always be in the thermodynamical dissolution/ionic area. pH can be adjusted with different acid concentrations and liquid to solid ratios.
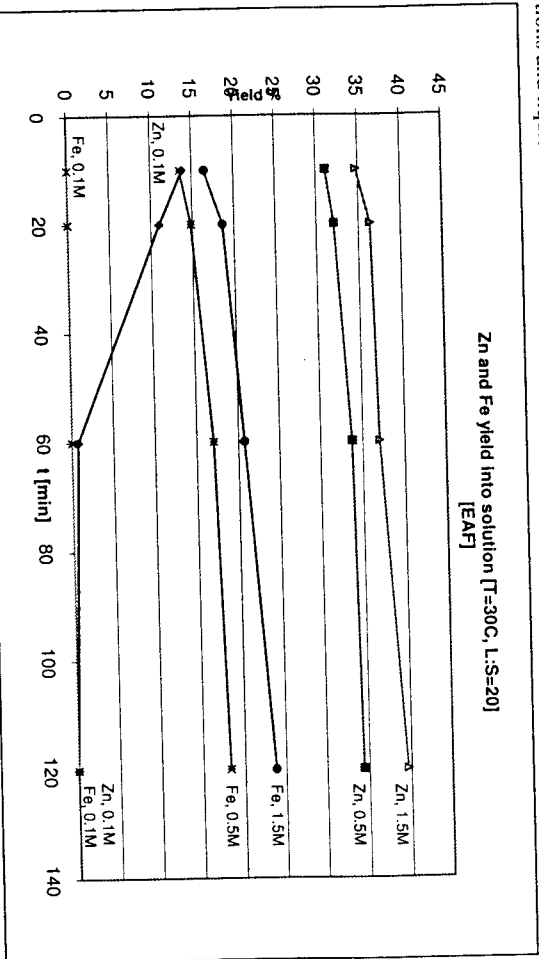
Figure 3: Zn and Fe yield into solution, effect of acid concentration, T = 30 °C, L:S = 20

3.1.2 Effect of liquid to solid ratio

Figure 4 shows the effect of L:S ratio and acid concentration on iron extraction in T = 30 °C for 0.1 M – 1.5 M. With 0.5 M sulphuric acid concentration and L:S = 10, the iron concentration in the solution starts to decrease as iron precipitates probably as hydroxides. With 1.5 M and L:S = 10 or 20 the same decreasing effect of Fe concentration in solution phase is not observed as free acid is available. Here, the difference between L:S = 10 or 20 is not significant. In the lowest 0.1 M acid concentration, iron stays in solid residue completely with both L:S = 10, 20. Considering this, the

L:S ratio is observed to have an effect, together with the acid amount, on adjusting the pH to an area where the metal extraction is possible.
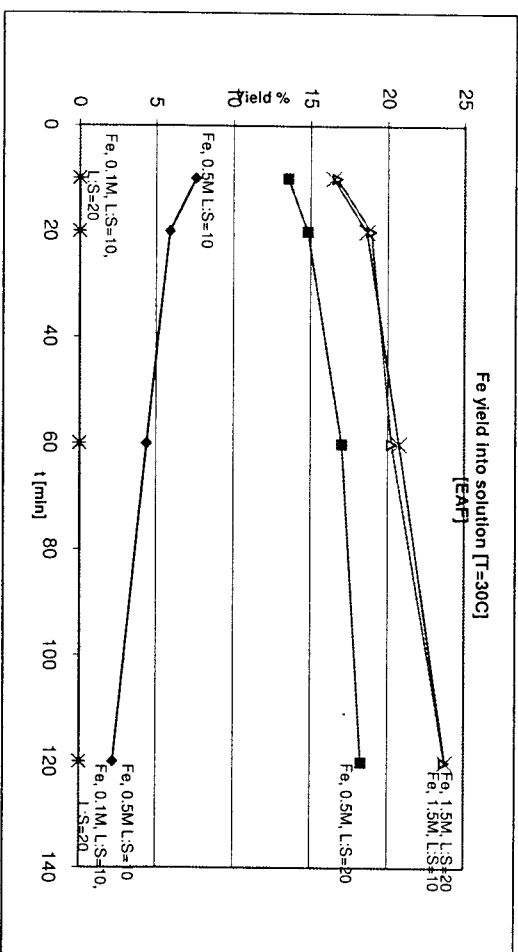
Figure 4: Fe yield into solution, effect of L:S ratio and acid conc., T = 30 °C, 0.1 M – 1.5 M

3.1.3 Maximum yield and the best selectivity

Maximum Zn dissolution of 60 – 70 % was achieved with 3 sets of parameters: 1.5 M – 90 °C – L:S = 20, 1.5 M – 90 °C – L:S = 10 and 1.5 M – 60 °C – L:S = 10 after 120 min (the end point). At the same time however, around 8 % of Cr, 25 – 50 % of Ni and 40 – 70 % of Fe was extracted (Figure 5) into solution phase. Mo was almost totally extracted (not in figure). This result is not satisfying as Zn amount in the solids should be as low as possible, i. e. 0.1 wt.-%, and the valuable elements as high as possible for recycling them back to process furnaces. Zinc is considered to be harmful material in steel production because it adds up in the furnace and shortens its life.
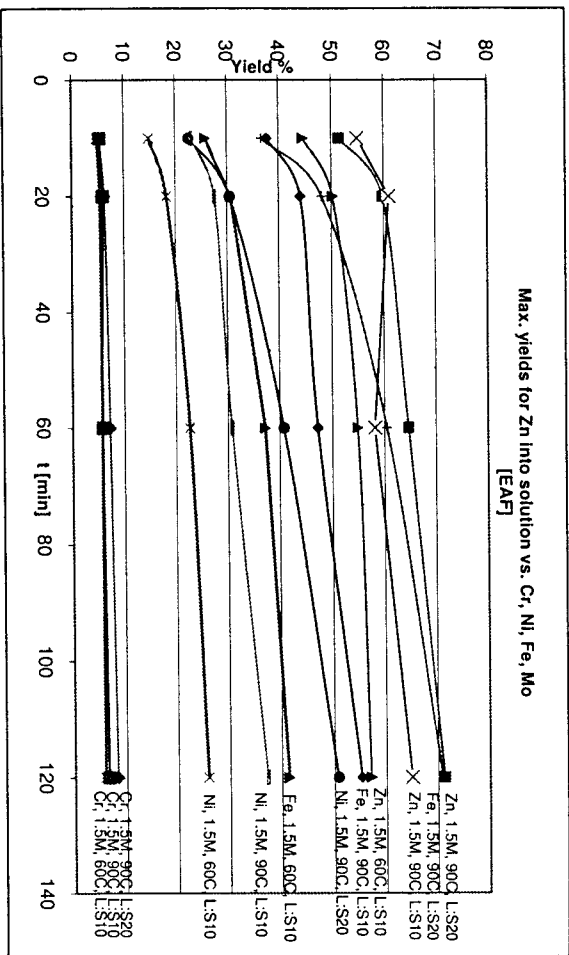
Figure 5: Maximum Zn yields into solution vs. Cr, Ni, Fe (Mo 100 %)

The highest Zn selectivity instead, was achieved with 0.5 M – L:S = 10 in temperatures 30 °C and 90 °C after 120 min. However, only around 33 – 36 % of Zn and at the same time 0 – 10 % of Cr, Fe, Ni and Mo were extracted (Figure 6). Mo shows decreasing of yield during 10 – 120 min maybe because of precipitation back to solid form.

Considering the low amounts of Zn extracted, there is probably not enough free acid to dissolve the hardly soluble zinc - ferrite structure from this particular EAF dust. Stronger acid or higher L:S ratio in the beginning is probably required for more zinc and iron to dissolve from the zinc - ferrite phase. Then subsequent and careful increase of pH to precipitate iron from the solution needs to be considered to achieve better selectivity for Zn. In addition, the dust formation and Zn – Fe phase formation conditions in the furnace should be researched in more detail and tests done from different spots of the dust formation line to see if it affects the amount of Zn – Fe produced and by that, Zn leachability afterwards.
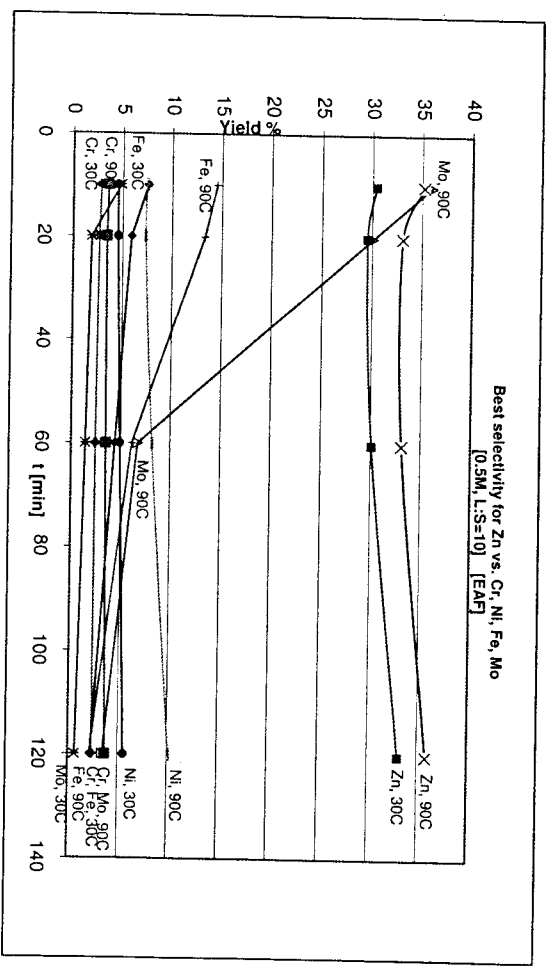
Figure 6: The best selectivity parameters for Zn vs. Cr, Ni, Fe, Mo, 0.5 M, L:S = 10

3.2 AOD dust

3.2.1 Effect of temperature

The same effect of temperature on extraction yield kinetics is seen also with AOD dust (Figure 7). Higher temperature will leach Zn and Fe faster.
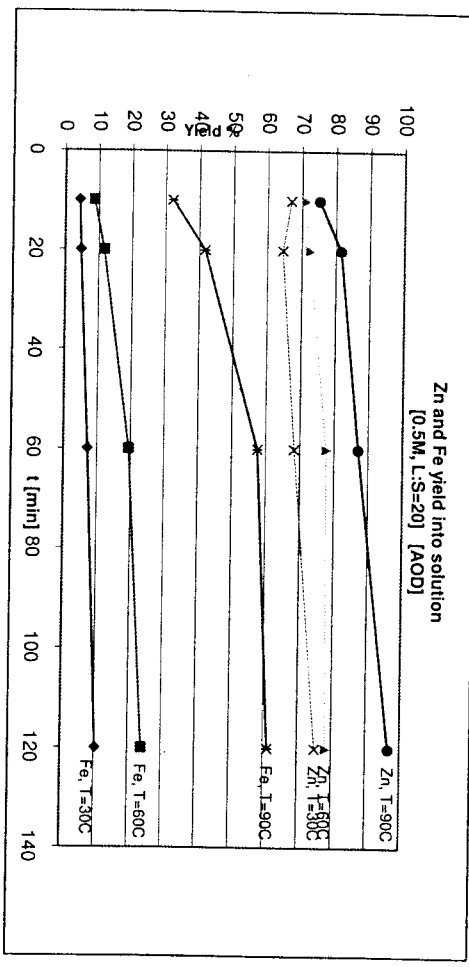
Figure 7: The effect of temperature on zinc and iron dissolution, 0.5 M acid, L:S = 20

3.2.2 Effect of L:S ratio and acid concentration

The effect of liquid to solid ratio and acid concentration on AOD dust is presented in Figure 8.

Zn and Fe yield into solution [T=30C] [AOD]

(Yield % vs t [min]; legend: Zn, 1.5M, L:S=10; Zn, 1.5M, L:S=20; Zn, 0.5M, L:S=20; Zn, 0.5M, L:S=10; Zn, 0.1M, L:S=20; Fe, 1.5M, L:S=10; Fe, 1.5M, L:S=20; Fe, 0.5M, L:S=20; Fe, 0.5M, L:S=10; Fe, 0.1M, L:S=10,20)

Figure 8: Zn and Fe yield into solution, effect of L:S ratio and acid concentration, T = 30 °C

A fast dissolution for Zn is observed after 10 or 20 min for 0.5 M and 1.5 M – L:S = 10, after which the yield decreases to increase again. This is probably due to a fast dissolution of easily soluble ZnO phase. Questionable is why does the yield seem to increase again after decreasing. This could be due to a slower dissolution mechanism of other Zn phases in the AOD dust and before that a precipitation of already dissolved Zn. However, for 1.5 M – L:S = 20 this drop of Zn yield does not occur. This could be because of sufficient free acid is available to continue the dissolution of Zn. Remaining question is why does the dissolution of Zn is higher for 0.5 M than 1.5 M in the beginning. There should be more tests from 0 – 10 min to see if 1.5 M will dissolve zinc more rapidly during that period and then starts to precipitate to be extracted again (Figure 8).

Iron instead, is slowly and linearly dissolving towards 120 min, except 0.1 M in which it remains in the solid phase. There are no major differences between L:S = 10 or 20, except for Zn in 0.1 M where it seems that higher amount of free acid (L:S = 20) is enough to create low pH to dissolve zinc contrary to L:S = 10 where Zn start to precipitate from 10 min to 120 min as pH increases slowly during the experiment.

3.2.3 Maximum yield and the best selectivity

The maximum Zn extraction of around 95 % were achieved with 3 parameter groups: 1.5 M – 90 °C – L:S = 10, 0.5 M – 90 °C – L:S = 20 and 1.5 M – 60 °C – L:S = 10 after 120 min. And with the same parameters, Cr was extracted around 10 %, Ni 25 – 45 %, Fe 35 – 55 % and Mo 65 – 85 % into the solution phase (Figure 9). For the solids to be recycled, almost all zinc should be leached and then precipitate iron preferably as hematite and see if Ni, Cr, and Mo will precipitate at the same time.

Max. yields for Zn into solution vs. Cr, Ni, Fe, Mo [AOD]

(Yield % vs t [min]; legend: Zn, 1.5M, T=90C, L:S=10; Zn, 0.5M, T=90C, L:S=20; Mo, 0.5M, T=90C, L:S=20; Mo, 1.5M, T=60C, L:S=10; Mo, 1.5M, T=90C, L:S=10; Ni, 1.5M, T=90C, L:S=10; Fe, 0.5M, T=90C, L:S=20; Fe, 1.5M, T=60C, L:S=10; Fe, 1.5M, T=90C, L:S=10; Cr, 1.5M, T=90C, L:S=10; Cr, 1.5M, T=60C, L:S=10; Cr, 0.5M, T=90C, L:S=20)
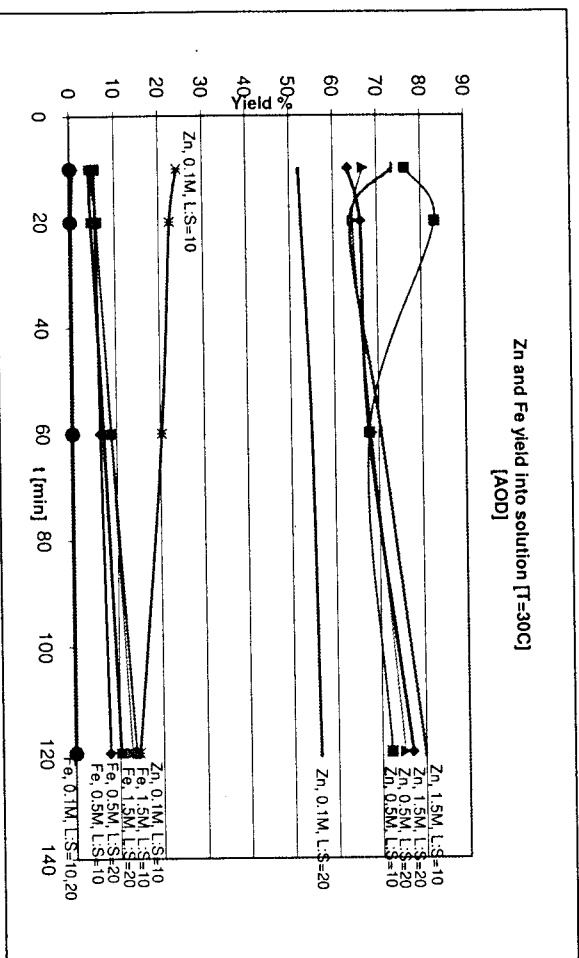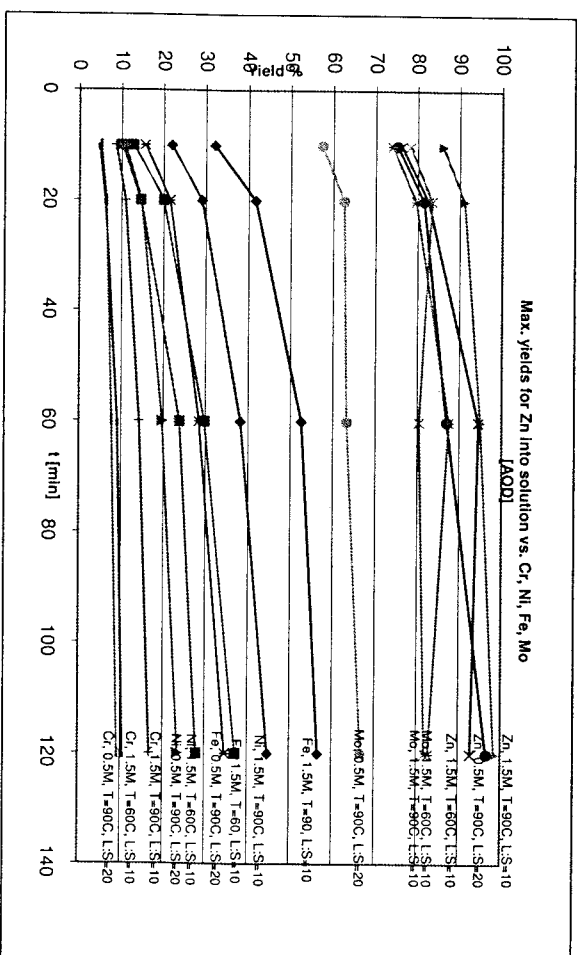
Figure 9: Maximum Zn yields into solution vs. Cr, Ni, Fe, Mo

The highest zinc selectivity instead, was achieved already after 20 min with 0.5 M – L:S = 10 in T = 30 °C and 90 °C. Around 80 % of Zn was extracted and at the same time Cr was extracted around 5 %, Ni around 10 %, Fe 8 – 15 % and Mo 20 – 60 % (Figure 10).

The effect of chemical and mineralogical composition is seen when comparing AOD and EAF leaching results. Where EAF dust consists of 13.1 wt.-% Ca, AOD dust has only 4.8 wt.-% of Ca. Zinc and iron amount varies also considerably between the dusts, as seen in Table 1. The lower amount of Ca in AOD dust suggests that the pH is lower during the experiment than in the EAF case for the same test parameters used and subsequently, the leaching result will be different. The mineralogy between these two dusts differs also, which is seen on another EMC 2011 publication: "Chemical and structural characterization of steelmaking dust from stainless steel production" (F. Kukurugya, T. Havlik, H. Makkonen, 2010). The mineralogy results indicate that hardly insoluble zinc - ferrite phases are very few or they do not exist in the AOD dust, but they are observed in the EAF dust mineralogy analysis. These facts indicate that maximum Zn dissolution yield and selectiv-

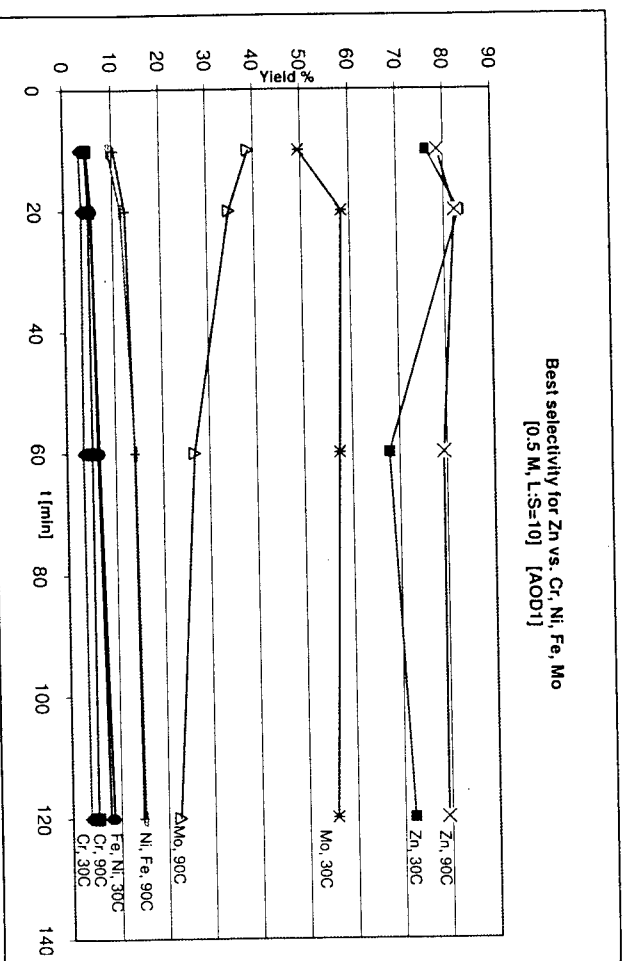ity for Zn will be better for AOD dust than for EAF dust. This was actually shown in the experiments.

**Best selectivity for Zn vs. Cr, Ni, Fe, Mo
[0.5 M, L:S=10] [AOD1]**

Figure 10: The best selectivity parameters for Zn vs. Cr, Ni, Fe, Mo, 0.5 M, L:S = 10

4 Summary

Leaching tests for stainless steel EAF and AOD production dusts were done in 0.1 M, 0.5 M and 1.5 M sulphuric acid concentrations and in temperatures 30 °C, 60 °C and 90 °C with liquid to solid ratios of 10 and 20 in atmospheric pressure. The main objective at this first stage was to i) maximize zinc extraction into solution and ii) selectively leach maximum amount of zinc leaving the valuable elements Cr, Ni, Fe, Mo in the solid residue for further treatment and possibly recycling back to stainless steel process without harmful Zn.

Increasing temperature and acid concentration were noticed to increase the dissolution kinetics. The L:S ratio together with acid concentration affects the pH value area during experiments which have influence on the dissolution and precipitation characteristics of the elements. Fe was found to precipitate with EAF dust in 0.5 M, T = 30 °C and 90 °C, L:S = 10.

For EAF dust, maximum Zn dissolution of 60 – 70 % was achieved with 1.5 M – 90 °C – L:S = 10 and 20 and 1.5 M – 60 °C – L:S = 10 parameter groups after 120 min. At the same time however, around 8 % of Cr, 25 – 50 % of Ni and 40 – 60 % of Fe was extracted and Mo almost totally extracted into the solution phase. The highest Zn selectivity from EAF dust was achieved with

0.5 M – L:S = 10 in temperatures of T = 30 °C and 90 °C after 120 min. However, only around 33 – 36 % of Zn and at the same time 0 – 10 % of Cr, Fe, Ni and Mo were extracted. In the AOD dust case, the maximum Zn extraction of around 95 % were achieved with 1.5 M – 60 °C and 90 °C – L:S = 10 and 0.5 M – 90 °C – L:S = 20 after 120 min. With the same parameters, Cr was extracted around 10 %, Ni 25 – 45 %, Fe 35 – 55 % and Mo 65 – 85 % into the solution phase. The highest zinc selectivity for the AOD dust was achieved already after 20 min with 0.5 M – L:S = 10 in T = 30 °C and 90 °C. Around 80 % of Zn was extracted and at the same time Cr was extracted around 5 %, Ni around 10 %, Fe 8 – 15 % and Mo 20 – 60 %.

One reason for the maximum yield and selectivity yield differences for Zn between EAF and AOD dusts is the difference in alkaline Ca concentration. It is probable that more free acid is needed for higher Zn yields if alkaline Ca containing phases consuming the acid are more predominant, like in EAF vs. AOD case. Second reason is probably the existence of more hardly soluble Zn-Fe phases over soluble ZnO phases in EAF dust than in AOD dust. However, at some stage, Zn should be removed almost completely from the solids for them (Cr, Ni, Mo, Fe) to be recycled back to process. Further steps after this study are the analysis of the solid residue (Cr, Fe, Ni, Mo) and a possible recovery of valuable metals like Ni, Mo or Zn also from the liquid phase with solvent extraction or ion exchange.

In the future, detailed tests with pH adjustments into iron precipitation area could possibly be made by adding more alkaline dust or acidic pickling process salt into the leaching reactor during operation. In addition, the dust formation and especially the zinc-ferrite formation conditions in the furnace should be researched in more detail. Tests should be done from different spots of the dust formation line, if possible, to see if it has an effect on the total amount of zinc-ferrite in the dust and, by that, Zn leachability afterwards.

Acknowledgements

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