Pressure Leaching of EAF Dust with Sulphuric Acid

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Hydrometallurgical pressure processing of EAF steelmaking dusts (by Paul Wurth S.A., Luxembourg) is investigated on a laboratory scale in the temperature range of 100 to 250 °C. The behaviour of zinc, iron and calcium under the influence of sulphuric acid as the leaching agent is discussed. The effect of temperature/pressure on the yield of the Zn-extraction is small. Only a relative small increase of pressure up to 4,1 bar at 150 °C shows some effect. The increase of temperature/pressure decreases the iron yield in a prepared solution. The concentration of H_2SO_4 is the most important factor for the increase of the Zn-extraction. After one or two days, the calcium sulphate generated in the solution is precipitated during the cooling of the solutions that were leached under ambient conditions. This does not apply to solutions obtained by leaching at higher temperatures/pressures. In regard to the selectivity of leaching, the optimal parameters are: T = 150 °C, c = 0.4 mol/dm^3 H_2SO_4 and an agitating speed of 700 rpm.

Keywords:

Hydrometallurgy – EAF dusts – Zinc – Iron – Pressure leaching – Sulphuric acid

Schwefelsaure Drucklaugung von Elektrolichtbogenofenstaub

Zur Rückgewinnung des Zinks wurde Elektrolichtbogenofenstaub aus der Stahlproduktion von Paul Wurth, Luxembourg, hydrometallurgisch unter hohem Druck und im Temperaturbereich zwischen 100 °C and 250 °C behandelt. Das Verhalten von Zn, Fe und Ca in Schwefelsäure als Laugungsmittel wird diskutiert. Der Druck- und Temperatureinfluss auf die Zn-Extraktion erweist sich als gering. Lediglich eine moderate Steigerung der Temperatur auf 150 °C (Druckanstieg auf 4,1 bar) beeinflusst das Lösungsverhalten in geringem Maße positiv. Eine Druck- und Temperatursteigerung verringert dagegen die Auflösung des Eisens signifikant. Die Konzentration der Schwefelsäure ist der wichtigste Faktor bezüglich der Steigerung der Zn-Extraktion in der Lösung. Im Unterschied zu den Lösungen, die unter Hochdrucklaugungsbedingungen hergestellt werden, wird Kalziumsulfat nach ein oder zwei Tagen aus den Lösungen, die unter Atmosphärendruck entstanden sind, abgesetzt. In Bezug auf die Selektivität der Laugung sind die folgende Parameter optimal: T = 150 °C, c (H_2SO_4) = 0.4 mol/dm^3 und eine Rührerdrehzahl von 700 U/min.

Schlüsselwörter:

Hydrometallurgie – Elektrolichtbogenofenstaub – Zink – Eisen – Drucklaugung – Schwefelsäure

La lixiviation à l'acide sulfurique et sous pression des poussières de four à arc

Lixiviación a elevada presión de polvos del horno de arco con ácido sulfúrico

1 Introduction

During the production of steel from scrap in electric arc furnaces a considerable degree of dust is recovered in gas cleaning units. In recent years the presence of leachable Zn, Pb, Cd, Cr and other metals within the dust has caused the material to be widely regarded as a toxic waste that is unsuitable for the disposal at landfills. For the same reason it is very complicated to use this waste as secondary raw material for the iron or steel production. Moreover, there is a growing need to process galvanised steel scrap from automobiles, washing machines, etc., in order to remove the zinc coating that otherwise causes problems in the remelting of the scrap in electric arc furnaces. There is a widespread presence of heavy metals in steelmaking dusts, for example 2 to 30 % Zn, 0.3 to 6 % Pb, 0.01 to 0.2 % Cd, etc. Moreover, a high amount of minor elements can be found such as Al, As, Ba, Ca, Cl, Cr, Cu, Hg, K, Mg, Mn, Mo, Na, Ni, P, Se, Si, Ti, Zr [1].

The mineralogical analysis shows the presence of iron in oxidic form, mostly as hematite Fe_2O_3, magnetite Fe_3O_4 and ferrite as franklinite $ZnFe_2O_4$. Zinc can be found mostly in two basic compounds, namely as oxide ZnO and ferrite $ZnFe_2O_4$, possibly complex ferrite, for example $(ZnMnFe)_2O_4$. The nature of the zinc presence seems to be the basic indicator for an effective method of the EAF dust treatment. ZnO in principle is the form easily workable both with pyro- and for the hydrometallurgical methods, but the ferrite form is considerable refractory. Moreover, it is almost impossible to predict the nature of the presence of the zinc compound. The negotiability of the chosen process is, of course, affected by the quantity of heavy metals occuring in the dust.

A number of hydrometallurgical processes has been developed in order to treat the dust from steel plants. Early efforts were directed towards technologies based on conventional sulphuric acid. Those processes failed due to the high Fe/Zn ratio of the EAF dusts and especially due to the high halogen content, which cannot be adjusted during the zinc sulphate electrolysis. An alkaline leaching offers the potential advantage that the iron remains largely insoluble, and the effort to develop alkaline leaching technologies for EAF dusts proceed. Such processes are limited, however, by their inability to recover zinc from zinc ferrite unless a reducing roast is carried out first. Both approaches result in halide-containing solutions. This kind of processing is difficult. Because of the high chloride content of EAF dusts, chloride-based processing technologies could offer several advantages.

In the seventies of the last century, AMAX operated a pilot plant based on caustic leaching, on the purification of solution by zinc dust and on electrowinning. This development was abandoned due to both technical and economic problems. A similar plant [2, 3], based on caustic leaching and known as the Cebedau process, was later built in France. The steelmaking dust containing 21.2 % of Zn and 3.6 % of Pb was used in this process.

Another hydrometallurgical process of steelmaking dust treatment is based on a diluted acetic acid leaching. This method dissolves the lime as calcium acetate and is followed by an ammonia leach that serves to remove the zinc from the residue [4]. The zinc is precipitated by CO_2 as basic zinc carbonate while the final residue is leached with hot acetic acid in order to remove the lead. The calcium acetate solution is reacted with sulphuric acid with the aim of producing gypsum and acetic acid. A similar research work is also published by Barret et al. [5]. A steelmaking dust containing 17.33 % of Zn, 2.09 % of Pb, and 0.083 % of Cd is used in this process.

The Zincex process [6] was originally developed in order to recover the zinc from the chloride-sulphate solutions. As applied to EAF dust, this approach serves to leach the dust in diluted sulphuric acid in order to solubilise the ZnO-component that is then treated directly by solvent extraction.

The Terra Gaia process is based on the dissolution of both ZnO and $ZnFe_2O_4$ in $FeCl_3$-HCl media at 175 °C [7, 8]. The steelmaking dust containing 18.5 % of Zn and 8.84 % of Pb is used in this process. The EAF dust is stirred by a ferrous chloride solution produced by the reaction of scrap iron with chlorine gas. It is then injected into an autoclave at a temperature of 175 °C. Oxygen is also injected resulting in the oxidation of $FeCl_2$ to $FeCl_3$, which hydrolyses extensively at 175 °C and also produces HCl. The generated hydrochloric acid attacks both the ZnO and $ZnFe_2O_4$ constituents, and any residual $FeCl_3$ reacts rapidly with ZnO. Presumably, the abundant Fe_2O_3 in the EAF dust passes through the autoclave largely unaffected.

A leaching test using acidified calcium chloride indicates that the selective leaching of zinc over iron could be achieved by employing ultrasound [9]. However, the zinc oxide-rich calcine is prepared by double-kiln pyrometallurgical process before leaching.

The leaching of EAF dust using a sulphuric acid solution (0.1 to 2.0 M) in the temperature range of 18 to 61 °C has been carried out by Cruells et al. [1]. The EAF-dust containing zincite ZnO and zinc ferrite is used for this leaching test. The sample containing 22 % Zn and 3 % Pb is used for these experiments.

The results reveal that for the zinc species the reaction rate is virtually independent of the sulphuric acid concentration in the range investigated. In contrast to that, for the iron species the reaction rate is a function of the acid concentration. The achieved zinc efficiency amounts to around 90 %, whereas the iron efficiency is within the range of 20 to 50 % depending on the acid concentration and the temperature. The total leaching time is one hour.

Jandova et al. [10] have leached steelmaking dust containing 14 % Zn and 1.7 % Pb. The zinc occurs mostly as zinc ferrite, while iron as magnetite and, of course, as ferrite. The zinc leaching efficiency is between 45 and 90 % at 80 °C depending on the sulphuric acid concentration that is used in the range 0.5 and 3 M. The iron efficiency is between 10 and 90 %, again depending on the sulphuric acid concentration.

Jha and Duyvesteyn [11] have explored the sulphuric acid leaching of US steel plant dusts, both at atmospheric and elevated pressures. The results show a zinc leaching efficiency of between 32 and 76 % by using the autoclave leaching (2 hours, 270 °C, 2 M H_2SO_4) and 53 to 79 % by using atmospheric leaching (4 hours, 70 °C, 2 M H_2SO_4). High leaching zinc efficiencies around 97 % are achieved when a high concentration of 4 M H_2SO_4 is used.

Lupi et al. [12] have leached EAF steelmaking dust containing a high proportion of zinc oxide (63.1 %) and being poor in zinc ferrite. This material is leached in 1.5 M sulphuric acid at 30 °C in order to provide an aqueous solution, being rich in Zn, Fe and Mn, and a sludge containing Pb and Ca sulphate. Depending on the H_2SO_4 concentration the extraction yield of Zn is between 73.9 % (0.5 M H_2SO_4) and 95.6 % (1.5 M H_2SO_4), while the iron extraction is between 47.3 % and 59.7 %.

The present study aims at exploring the possibility of securing the selective leaching of zinc (and other heavy metals) out of EAF dust. The main factors investigated are the effect of the total pressure depending on the solution temperature and the solid/liquid ratio on the dissolution of Zn, Pb, Ca and Fe when sulphuric acid as lixiviant is used.

2 Experimental

2.1 Material

The dust sample that is used was given by Paul Wurth S.A. and shows the following values in the chemical analysis (Table 1), determined by the ICP method.

Burning out of carbon is mostly responsible for the loss of ignition. When taking into account the oxides of present elements almost 100 % is achieved in the chemical analysis. The sample of EAF dust is also examined by an X-ray diffraction qualitative phase analysis (Figure 1).

The results are shown in Figure 1, from which the presence of compounds such as franklinite Zn_2FeO_4, zincite ZnO, magnetite Fe_3O_4 can be inferred. Also cristobalite SiO_2 and complex franklinite $(ZnMnFe)(FeMn)_2O_4$ can be found. Other phases, which, on the basis of the chemical analysis, could be present (Table 1), are probable below the detection limit.

2.2 Experimental set-up and procedure

Leaching tests are performed in a 2 dm^3 autoclave, manufactured by the Autoclave Engineers, USA. The temperature is controlled within ± 1 °C by a temperature control system, manipulating both the electrical heating mantle and the water cooling system. Agitation is provided by a titanium impeller driven magnetically. The stirring speed is constant and amounts to 700 rpm. The autoclave is equipped with an acid injection device and a system to withdraw samples that was designed by the IME (Figure 2).

Leaching tests are performed with a solution of 900 ml of 0.4 M H_2SO_4. The sample weight is within the range 30 to 90 g. The acid-to-EAF-dust ratio a/d amounted between 0.4 and 1.2. The temperatures being used and the corresponding pressures are given in Table 2.

Fig. 2: 2 l-Ti autoclave at IME, RWTH Aachen

Tab 2: Leaching temperatures and pressures being used for the tests

Temperature [°C]	100	150	200	250
Pressure [bar]	1.0	4.1	14.5	41.0

The samples for elemental analysis are taken according to a fixed time-schedule after 2, 15, 30, 45 and 60 minutes of leaching. Due to fact that the pulp is taken from the autoclave, the samples are filtered into test-tubes and thus a clear solution is achieved. Varyingly, the solution can be colourless, weakly yellow, weakly emerald green or weakly blue.

In some samples (after filtering) the white precipitate of calcium sulphate builds up after one or more days. Yet, this never occurs immediately after the cooling.

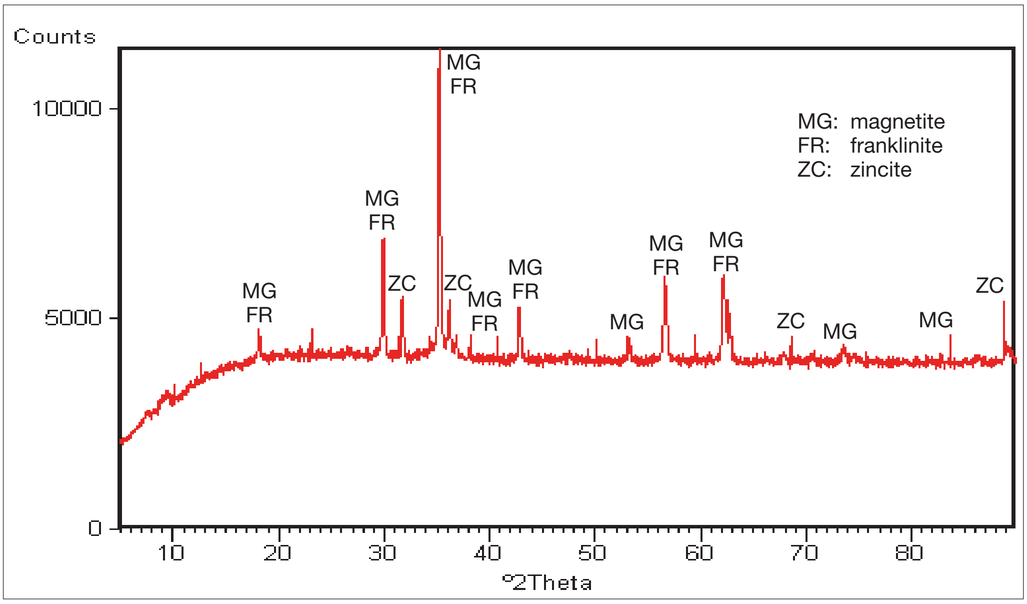

Fig. 1: XRD pattern of the Paul Wurth S.A. sample

Tab. 1: Chemical analysis of EAF dust by Paul Wurth S.A.

Element	Fe	Zn	Pb	Si	Ca	Mn	Cu	Cr	Ni	Ti	Cd	Sb	As	Cl^-	SO_4^{2-}	Loss of ignition
[%]	27.8	20.9	2.7	2.1	4.3	3.2	0.36	0.77	0.06	~0.04	0.027	0.069	< 0.1	0.89	0	12.18

The elementary analysis, carried out in order to search for Zn, Fe, Cd and Pb, is made by means of the ICP Plasma SpectroFlame EOP machine. All results that are achieved are re-calculated because of the change of the pulp volume due to the sampling.

3 Results and discussion

3.1 Mechanism of leaching of EAF dust with sulphuric acid

According to the results obtained in characterisation of EAF dust and thermodynamic study, the reactions of the main species occuring in the sample and their stoichiometry can be stated as follows:

$$ZnO + H_2SO_{4(aq)} \rightarrow ZnSO_{4(aq)} + H_2O \quad (1)$$

$$ZnFe_2O_4 + 4H_2SO_{4(aq)} \rightarrow ZnSO_{4(aq)} + Fe_2(SO_4)_3 + 4H_2O \quad (2)$$

$$ZnFe_2O_4 + H_2SO_{4(aq)\,)} \rightarrow ZnSO_{4(aq)} + Fe_2O_3 + H_2O \quad (3)$$

Although no positive proof for the presence of calcium compounds is found, we can predict its presence as CaO or $CaCO_3$ [4, 12]. Also $CaFe_2O_4$ or $Ca_2Fe_2O_5$ could be present. In this case the side reactions can occur:

$$CaCO_3 + H_2O_{4(aq)} \rightarrow CaSO_4 + CO_2 + H_2O \quad (4)$$

$$CaO + H_2SO_{4(aq)} \rightarrow CaSO_4 + H_2O \quad (5)$$

$$CaFe_2O_4 + 4H_2SO_{4(aq)} \rightarrow CaSO_4 + Fe_2(SO_4)_{3(aq)} + 4H_2O \quad (6)$$

$$Ca_2Fe_2O_5 + 5H_2SO_{4(aq)} \rightarrow 2CaSO_4 + Fe_2(SO_4)_{3(aq)} + 5H_2O \quad (7)$$

Calcium sulphate has a limited solubility and, according to the conditions, is located in aqueous solutions or in the solid residue. It is important to note that most of the iron remains in the residue. Reaction (2) occurs slowly at room temperature and runs at a high rate and at elevated temperatures. Unfortunately, the reactions (4) and/or (5) do take place at all sulphuric acid concentrations, which increases the sulphuric acid consumption.

3.2 Leaching studies with sulphuric acid at high temperature/pressure

As described above, the leaching tests are performed at various temperatures/pressures within the range of 100 to 250 °C that corresponds to 1 to 41 bars of total pressure in an autoclave. In Figure 3a to d the extraction curves at 0.4 M H_2SO_4 and various acid : dust (a/d) ratios for Zn at different temperatures/pressures are presented.

Temperature and pressure have little effects on the yield of the zinc extraction. Basically, only a relatively small increasing of pressure up to 4.1 bar and up to 150 °C respectively shows some effect on the zinc leaching kinetics as it is demonstrated in Figure 4a to c. It is valid for each studied solid : liquid ratio being studied.

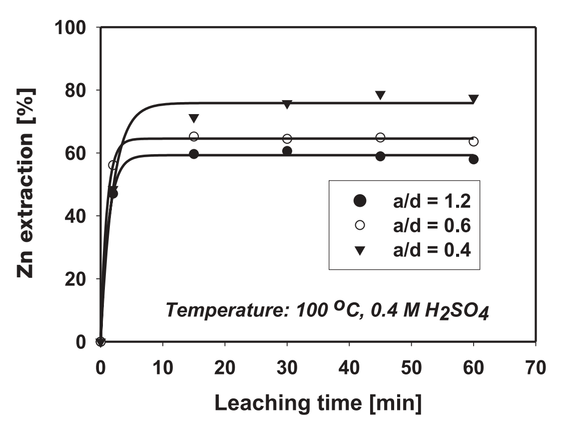

Fig. 3a: Extraction of Zn by sulphuric acid at a temperature 100 °C and 1 bar pressure

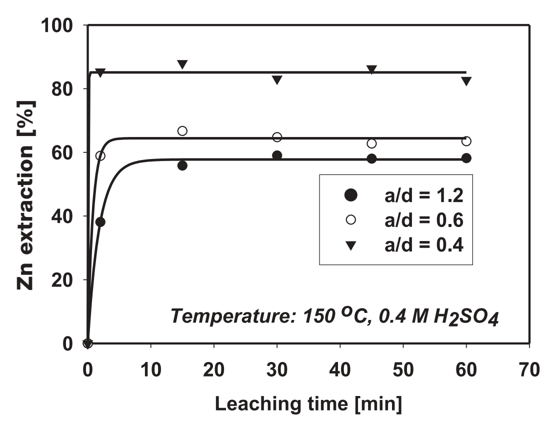

Fig. 3b: Extraction of Zn by sulphuric acid at a temperature 150 °C and 4.1 bar pressure

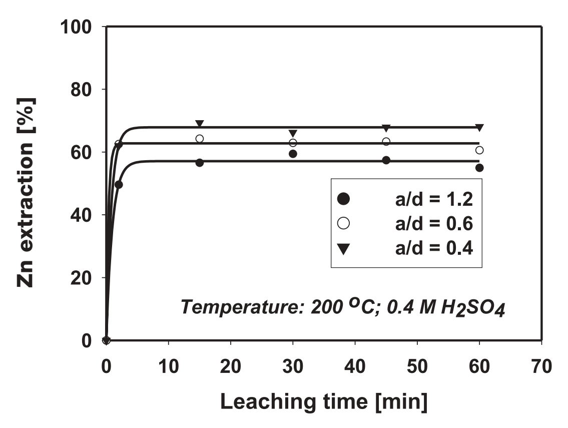

Fig. 3c: Extraction of Zn by sulphuric acid at a temperature 200 °C and 14.5 bar pressure

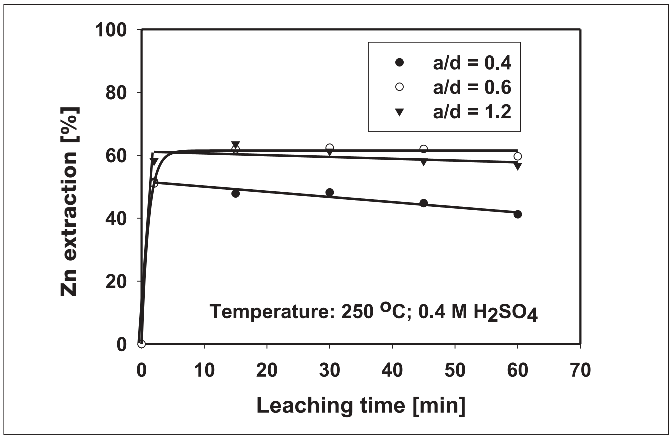

Fig. 3d: Extraction of Zn by sulphuric acid at a temperature 250 °C and 41 bar pressure

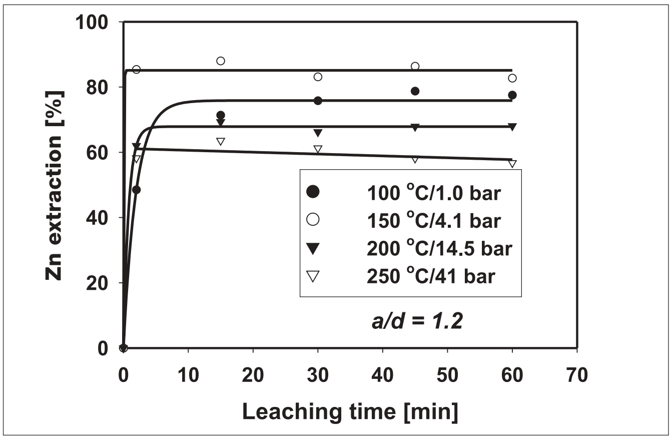

Fig. 4a: Extraction of Zn by sulphuric acid for 30 g charge and under various pressures

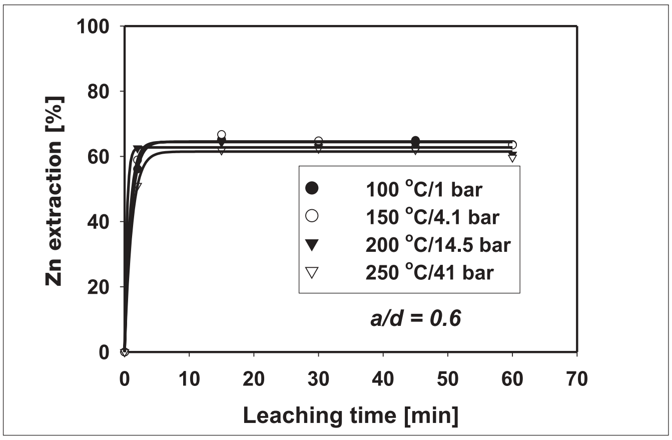

Fig. 4b: Extraction of Zn by sulphuric acid for 60 g charge and under various pressures

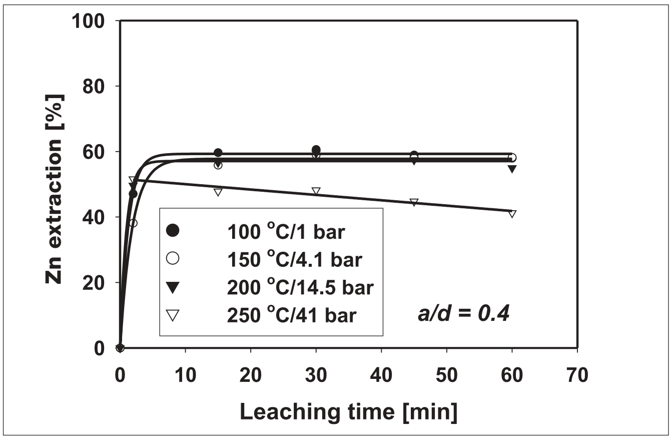

Fig. 4c: Extraction of Zn by sulphuric acid for 90 g charge and under various pressures

Since an increase of the quantity of the charge causes the reduction of the zinc yield, the concentration of sulphuric acid seems to be the most important factor for zinc extraction. The maximum value for Zn-extraction of $\alpha = 84$ % is obtained at a temperature of 150 °C, in a time of 10 min and with a concentration of sulphuric acid of $c = 0.4$ mol/dm^3 (Figure 3b). A further increase of temperature up to 250 °C leads to a decrease of the Zn-extraction of approx. 60 %. The high pressure hydrometallurgical recovery of Zn from EAF dust is feasible at a relatively high recovery yield, while iron mostly remains in the solid phase. This is due to the use of a low concentration of sulphuric acid. Higher temperatures and pressures relatively increase the zinc yield. Figure 5a to d illustrates the extraction curves for Fe under the same conditions. In this case, the increase of temperature/pressure causes the decrease of the iron yield.

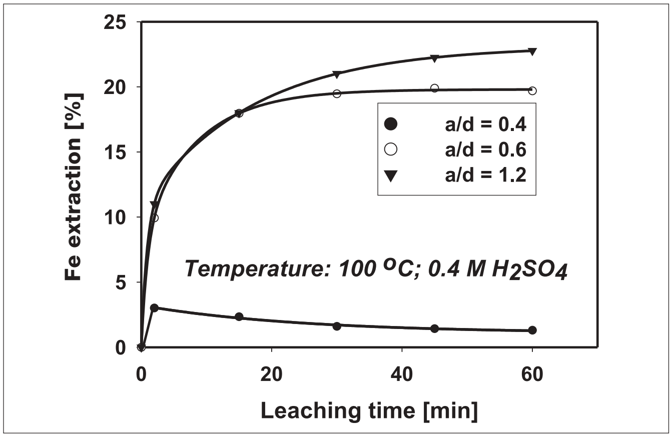

Fig 5a: Extraction of Fe with sulphuric acid at 100 °C temperature and 1 bar pressure

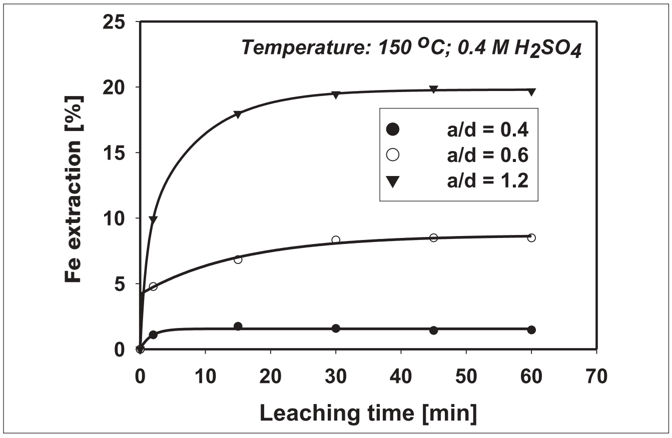

Fig 5b: Extraction of Fe by sulphuric acid at a temperature 150 °C and 4.1 bar pressure

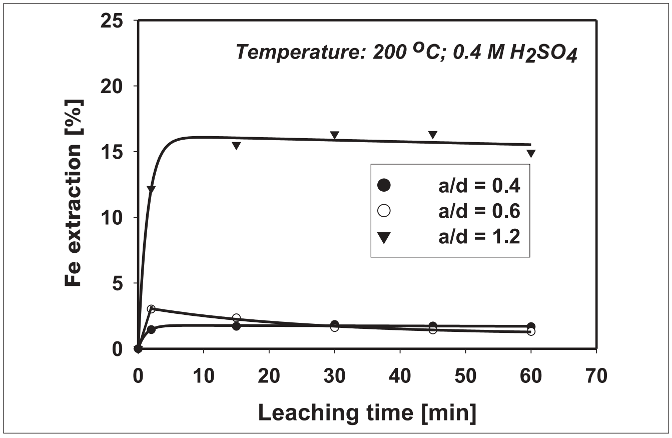

Fig 5c: Extraction of Fe by sulphuric acid at a temperature 200 °C and 14.5 bar pressure

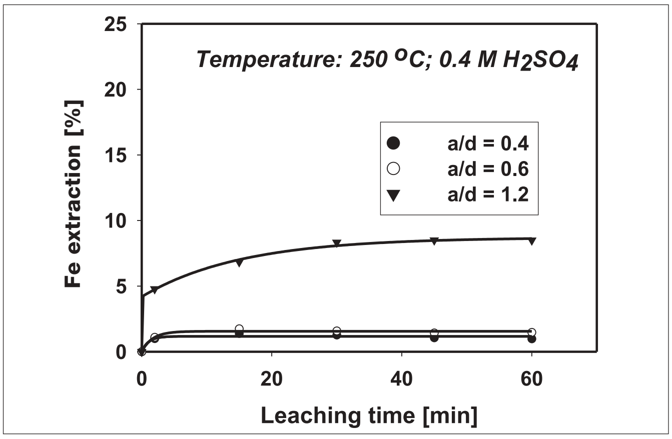

Fig 5d: Extraction of Fe by sulphuric acid at a temperature 250 °C and 41 bar pressure

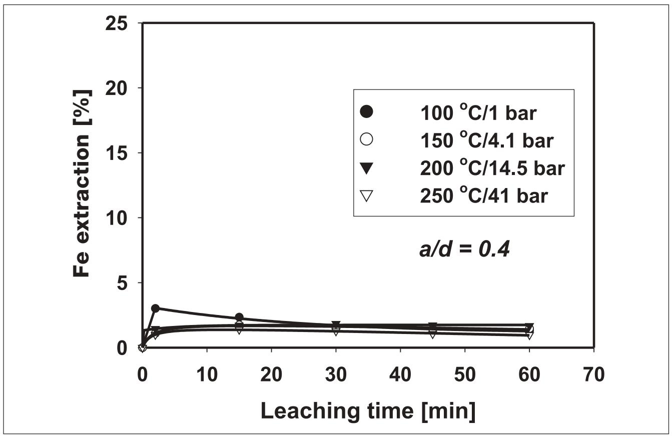

Fig. 6c: Extraction of Fe by sulphuric acid for 90 g charge and under various pressures

In such way almost the complete quantity of iron is able to precipitate while zinc is mostly transferred into the solution as it can be seen in Figure 6a to c.

It is observed that calcium sulphate is precipitated from the solution after the leaching and the cooling of the leaching solution, but not immediately subsequent to the leaching. It takes place slowly and the precipitate occurs after one to two days. It is interesting that it occurs only when a leaching temperature of 100 and 150 °C is applied. At higher temperatures/pressures the leaching calcium stays in soluble form basically forever.

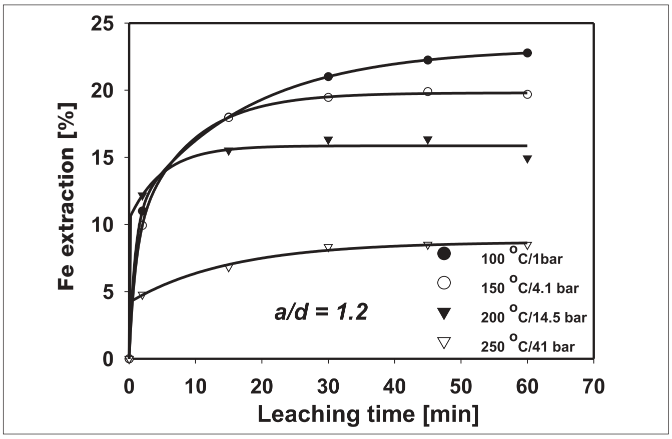

Fig. 6a: Extraction of Fe by sulphuric acid for 30 g charge and under various pressures

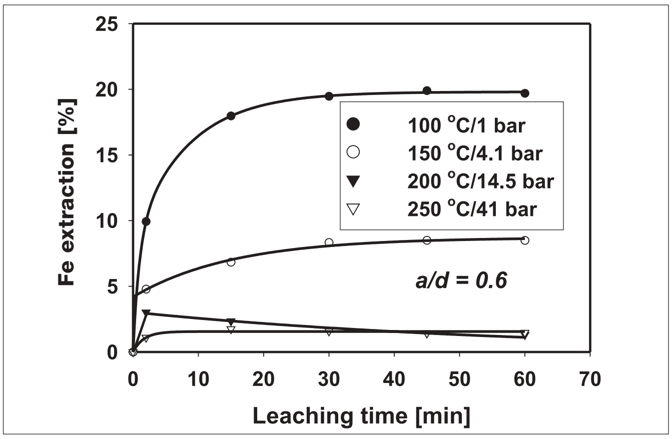

Fig. 6b: Extraction of Fe by sulphuric acid for 60 g charge and under various pressures

It is well known that, compared to the alkaline leaching, the acid leaching of EAF dust results in fast kinetics and relatively high metal leaching efficiencies. Unfortunately, also iron is considerably leached in the acid medium. This is the reason why the idea of finding an optimum solving during the acid leaching has been another aspect of this work.

The various concentrations of H_2SO_4 used in these experiments result mostly from thermodynamic considerations and calculations. The stability area of Fe^{2+} ion in the system Fe-S-H_2O reaches the values up to pH = 1.6 at 100 °C and pH = 0.4 at 250 °C. The simple calculation shows that the complete amount of acid should be spent in regard to the leaching of non-ferrous metals occuring within the EAF dust. In this way, probably a minimum of acid is spent for iron.

Figures 7a to b show the dependencies of acid behaviour during the leaching at boundary temperatures.

The final concentration of acid determined as free acid content within the leaching solution indicates the pH-values between 2 and 2.5 at 100 °C and 2 and 5 at 250 °C for the individual EAF dust charges. E-pH diagrams of the system Fe-S-H_2O show these pH values as out of range of the stability area of the Fe^{2+} ion. This fact reveals that iron must be precipitated into the solid rest. Figures 8a to b show the

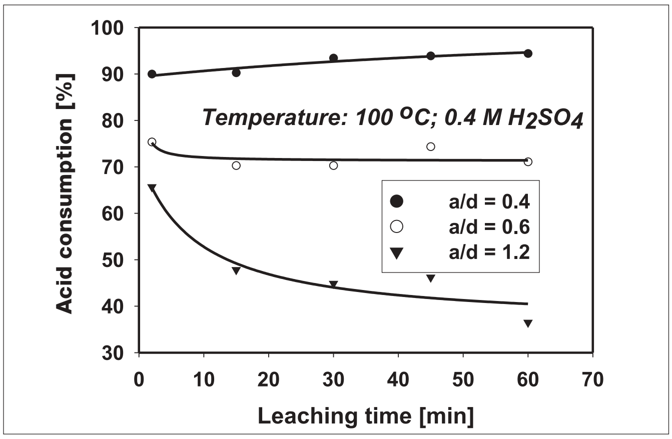

Fig. 7a: Acid consumption during the leaching at 100 °C and with various charges

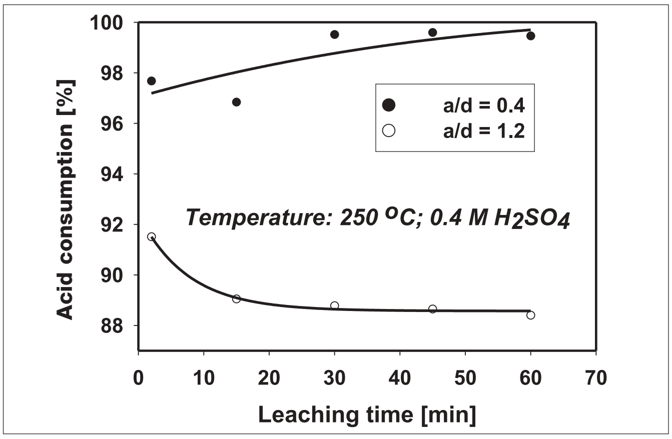

Fig. 7b: Acid consumption during the leaching at 250 °C and with various charges

correspondence between the acid consumption and the iron content in the solution during the leaching. For this reason this is in accord with the above-mentioned conclusion.

The precipitation of the white precipitate of calcium sulfate is observed in the leaching solution after approximately one day. However, it only occurs in solutions of lower temperatures/pressures used for the leaching, i.e. at 100 and 150 °C for each of the charges of the EAF dust being used. Although the thermodynamic study reveals that calcium sulfate should be stable at each temperature being applied and in the whole range of pH it was appears at the above-mentioned temperatures. The reason for this is not clear. To find out the reason can be the challenge for future investigation.

The results of the leaching with diluted sulfuric acid reveal at least two possibilities: It is possible to achieve a rather high Zn-extraction into the solution, but also a considerable Fe-content in the solution is manifest. This fact could cause a problem on the next step of the treatment of the solution. Also the iron yield for future recycling will be smaller. This situation is given when high a/d ratio is used.

On the other hand using a low a/d ratio for the leaching also means that the Zn-extraction is lower, but virtually no iron is transferred into the solution. Among these limitations the ideal leaching conditions are found.

Figure 9a to b show the dependencies of metal extraction on the leaching temperature/pressure for the highest and

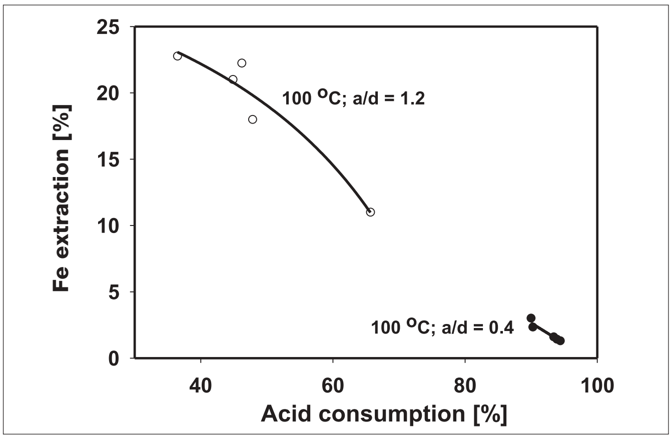

Fig. 8a: Acid consumption vs iron extraction dependence at 100 °C

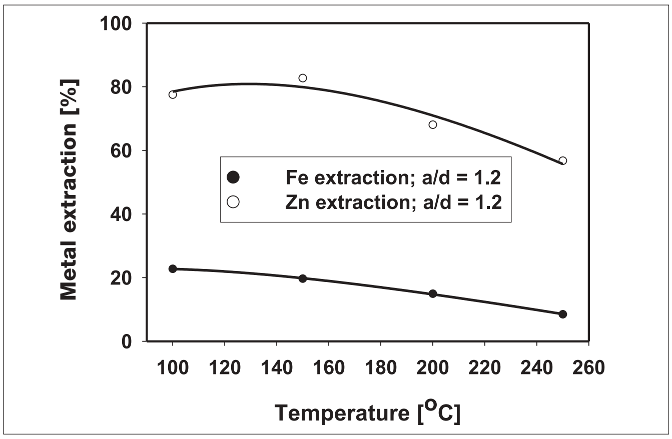

Fig. 9a: Metal extraction vs leaching temperature dependence at a/d = 1.2

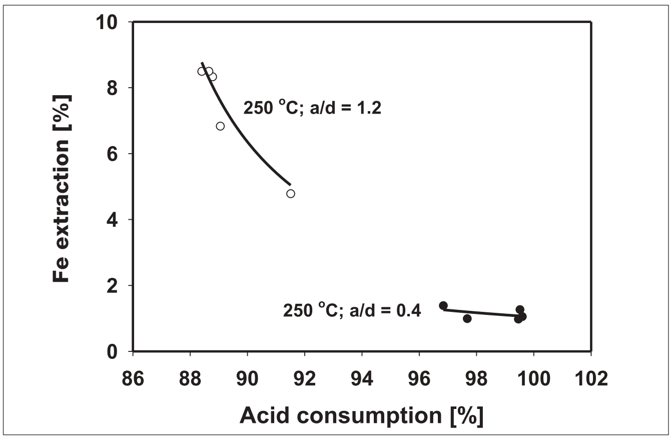

Fig. 8b: Acid consumption vs iron extraction dependence at 250 °C

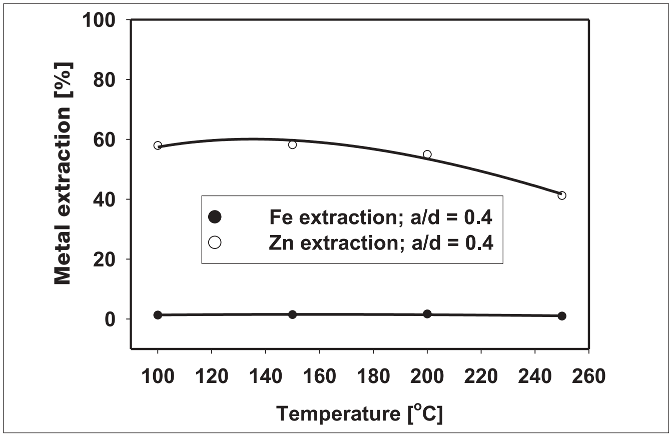

Fig. 9b: Metal extraction vs leaching temperature dependence at a/d = 0.4

Tab. 3: Metal extraction vs leaching temperature dependence at two a/d ratios

T [°C]	a/d = 1.2	a/d = 0.4				
	Fe extraction [%]	Zn extraction [%]	Zn/Fe	Fe extraction [%]	Zn extraction [%]	Zn/Fe
100	22.8	77.5	3.4	1.3	58.0	44.4
250	8.5	56.7	6.7	1.0	41.2	42.2

lowest dust charge, which corresponds to the amount of acid available for leaching. Table 3 shows the numerical values from Figure 9a to b.

Table 3 indicated that, at the maximal investigated a/d ratio of 1.2, the increase of temperature raises the leaching selectivity. This fact is not characteristic in regard to the analysis of the low ratio of a/d = 0.4. Unfortunately, it seems that higher values of selectivity are obtained by lower a/d ratios.

4 Conclusions

According to the results obtained in this work, the high pressure hydrometallurgical recovery of zinc from EAF dust is feasible at a reasonable recovery yield, while iron mostly remains in the solid phase. The latter is due to the use of a low concentration of sulphuric acid. The maximal value of selectivity is obtained by the minimal chosen a/d ratio of 0.4. At the maximal investigated a/d ratio of 1.2 the increase of temperature raises the leaching selectivity. On the other hand, higher temperatures and pressures moderately increase the zinc yield. In such way it is possible to set up the conditions for further electrolysis of the obtained solution by choosing the optimal leaching temperature/pressure and sulphuric acid concentration of EAF dust in order to achieve an optimum zinc yield into solution without an iron and calcium dissolution. However, the problem caused by the fact that the chemical and mineralogical composition of each steelmaking dust is individual makes it necessary to study the conditions of processing for each one.

Acknowledgement

The authors wish to express their gratitude to DAAD and Slovak grant agency VEGA (Grant 1/9379/02) for their financial support. This work was performed at IME Institute for Process Metallurgy and Metal Recycling of the RWTH University in Aachen.

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Receipt of manuscript: February 2, 2004