

Lead extraction in low-temperature dust roasting during metal scrap melting in electric arc furnace together with ammonium chloride

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Processing of zinc-containing dust from conventional steel making processes is rather actual problem and causes necessity to research and develop the new technologies. Possibility of selective lead extraction via low-temperature roasting of preliminary separated non-magnetic fraction of zinc-containing dust together with ammonium chloride is shown in this work. Based on the results of thermodynamic calculations of Gibbs free energy for interaction reactions between oxides of lead, zinc and iron with NH_4Cl , possibility of selective lead extraction from dust in the form of its chloride is displayed. The results of thermodynamic analysis are confirmed by the data of laboratorial experiments, when influence of temperature and chloride ammonium consumption on degree of lead sublimation was examined. Dramatic lowering of lead content in a cinder was revealed within the temperature range from 300 °C to 500 °C. The minimal lead content in a cinder was 0.18 %, it was reached at 600 °C. Further temperature rise has slight effect on decrease of lead content in a cinder. Optimal technological parameters were established: temperature – 500 °C, NH_4Cl consumption – the value exceeding its stoichiometric required amount (SRA) for lead chlorination by 1.5 times, roasting duration – 60 min. A cinder with minimal lead content with the following composition, % (mass.): ~49 Zn; 0.2 Pb; 19 Fe; 8 SiO_2 and other, was obtained for the optimal technological parameters. Lead extraction in fumes was high, more than ~97 %. The obtained results will be used in building of the complex technology for processing of zinc-containing dust and other products with similar composition from iron and steel enterprises.

Key words: zinc-containing dust, temperature, Gibbs free energy, roasting, chlorination, ammonium chloride, lead, zinc, extraction, fumes, cinder.

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Introduction

Up to 3 % of raw materials, which are introduced in a steel making furnace, transform in dust; this value can reach 10 % for several technologies [1, 2]. At present time, the volumes of accumulated dust at the iron and steel works are considered as critical and require maximal attention to their recycling. Especial actual problems are connected with accumulated zinc-containing dusts, which are characterized by multi-component chemical and physical compositions. Necessity of processing of steelmaking dusts concludes in the fact that they contain valuable metals. Average zinc content in these dusts is assessed at the level 8–10 %. However, complicated composition of such dust (% (mass.), up to 18 Zn, 30–60 Fe, 8–12 Ca, up to 6 Pb), which is typical for the iron and steel works in Kazakhstan, restricts its processing via conventional technologies. Especial difficulties are connected with selective zinc separation from lead. The attempts of recovery of zinc-containing dust for processing in metallurgical production lead to increase of zinc content in finished products (cast iron, steel, ferroalloys) and to lowering of their quality. High zinc content during melting pro-

cesses leads to formation of lead-containing dusts, which are harmful to health and environment, respectively, to violation of technological procedures in furnace operating.

The global annual summarized increase of zinc-containing dust amount is evaluated as 10–15 mln t. This tendency is also valid for the iron and steel works in Kazakhstan, where gross emissions of such dusts is observed in addition to dust accumulation. E.g., gross emissions of contaminants in the environment by production facilities of Arcelor Mittal company made 233 t in 2020, what exceeded 2019 value by 62.2 %.

Activity of average and small production enterprises in the field of iron and steel scrap processing (pipes, rails etc.) also provides substantial input in dust forming and accumulation. Such enterprises are not ready to apply wasteless technologies, so they can't sell dust due to its low quality and are forced to accumulate it in significant amounts.

A lot of various high-temperature technologies, directed on efficient processing of zinc-containing dust, are described in the technical literature [3–8]. However, majority of them remained at the stage of laboratorial researches, owing to necessity of large investments and power consumption, as well as low productivity. In addition, well-known pyrometallurgical methods (Waeltz process, fuming process) don't

provide obtaining of pure zinc fumes due to high lead amount in these fumes.

From the point of view of ecological safety, hydrometallurgical processes are considered as one of prospective technological directions [9–15]; they allow to provide selective extraction of valuable metals with possibility of high process automation and varying of technological parameters. However, they also have their disadvantages, such as high Fe content in solution, which seriously deteriorates quality of obtained products. Fe separation methods are also rather complicated and expensive processes.

Processing technologies for dusts and other technogenic wastes with use of chlorine-containing reactants (CaCl_2 , HCl etc) [16–18] present large interest. The developed technologies are based on use of high-temperature chloridizing roasting. Selective zinc separation from lead at high roasting temperatures (800–1100 °C) requires strict observance of the optimal technological parameters (temperature, consumption of chlorine-containing reactants, roasting time).

Brief analysis displays that the modern methods of dust processing don't allow to create profitable production for selective extraction of zinc, lead and other valuable components. As a result, most of metallurgical enterprises store this dust. So, search of a rational technology for dust processing is one of the key problems to be solved. Increase of lead content in dust (up to 6 %) restricts development of new high-efficient technologies for dust processing with extraction of zinc and other valuable metals.

General concept of the developing combined technology for complex dust processing via chlorination is directed on solving the existing problems and includes its dual-stage processing.

At the first stage magnetic separation provides fabrication of two products: magnetic and non-magnetic fractions. Magnetic fraction with high Fe content and minimal Zn and Pb content is directed on agglomeration before blast furnace practice. Non-magnetic fraction of dust, which was obtained after separation and is characterized by high content of zinc and lead, is subjected to further roasting during the second stage. This process includes obtaining of two products from magnetic fraction in presence of chlorine-containing substances for lead sublimation: (1) a cinder with high zinc content, which can be used as raw material for zinc production and (2) lead fumes, which are valid for use as raw material for lead production.

Based on general concept of the developing technology, influence of lead extraction from non-magnetic fraction of zinc-containing dust after scrap melting in electric arc furnace is examined in this research. This dust was obtained after magnetic separation, in the conditions of low-temperature roasting together with ammonium chloride.

Materials and methods of research

Zinc-containing dust, which was obtained during processing of secondary ferrous scrap (rails, pipes etc.) in electric arc furnaces at “Kazferrostal” company (Almaty) was used as initial product with the following content, % (mass.): 34.24 Zn; 4.12 Pb; 32.86 Fe; 5.3 SiO_2 and other.

Preliminary this dust was subjected to magnetic separation in the tubular magnetic analyzer 25T-SE. This device and technique of magnetic separation are described in details in the work [18]. The results of magnetic separation of zinc-containing dust displayed principal possibility of Fe extraction from initial dust. Fe-containing product (magnetic fraction) with high Fe content (up to 70 %) is obtained; it is rather suitable for steel making via the existing technologies.

The object of this research is non-magnetic fraction, which was obtained after magnetic separation of dust with its chemical and phase compositions presented in the **Tables 1 and 2**.

Table 1. Chemical composition of non-magnetic fraction

Initial sample	Content, % (mass.)					
	Zn	Pb	Fe	SiO_2	O	Other
Non-magnetic fraction	46.74	5.48	18.87	7.27	18.89	2.75

Table 2. Results of semi-quantitative X-ray phase of non-magnetic fraction

Substance	Chemical formula	Content, %
Quartzite	SiO_2	7.27
Franklinite	ZnFe_2O_4	23.14
Zincite	ZnO	50.74
Magnetite	Fe_2O_3	0.52
Litharge	PbO	5.91
Wustite	FeO	9.94

The studies were carried out using scanning electronic microscope and energy dispersive spectrometer JED-2300 (JEOL). Data processing from diffraction patterns and calculation of interplane distances were conducted using EVA software. Decryption of samples and search of phases were implemented via Search/match program with use of powder diffraction data PDF-2.

It was established that zinc in non-magnetic fraction of dust, which was obtained after magnetic separation, is presented mainly as oxide (50 %) and in the form of a stable compound – zinc ferrite (franklinite) with its content 23 %. Lead is presented as its oxide (~6 %).

The model of thermodynamic chlorination reactions for oxides of Pb, Zn and Fe by ammonium chloride in the conditions of low-temperature roasting (300–600 °C) was examined from the point of view of the established results of phase composition in non-magnetic fraction. Thermodynamic calculations were concluded in determination of Gibbs free energy loss depending on temperature variation using software HSC Chemistry 8.1.5, Outotec.

Experimental unit and technique

Roasting was carried out in the horizontal electric furnace Nabertherm 50/250/12 with thermal regulated controller B410. Alundum “boat-form boxes” with the size 120×60×20 mm were used as containers for charging of initial materials.

The scheme of laboratorial unit for examination of influence of NH_4Cl consumption and temperature on lead extraction from a dust non-magnetic phase is showed on the **Fig. 1**.

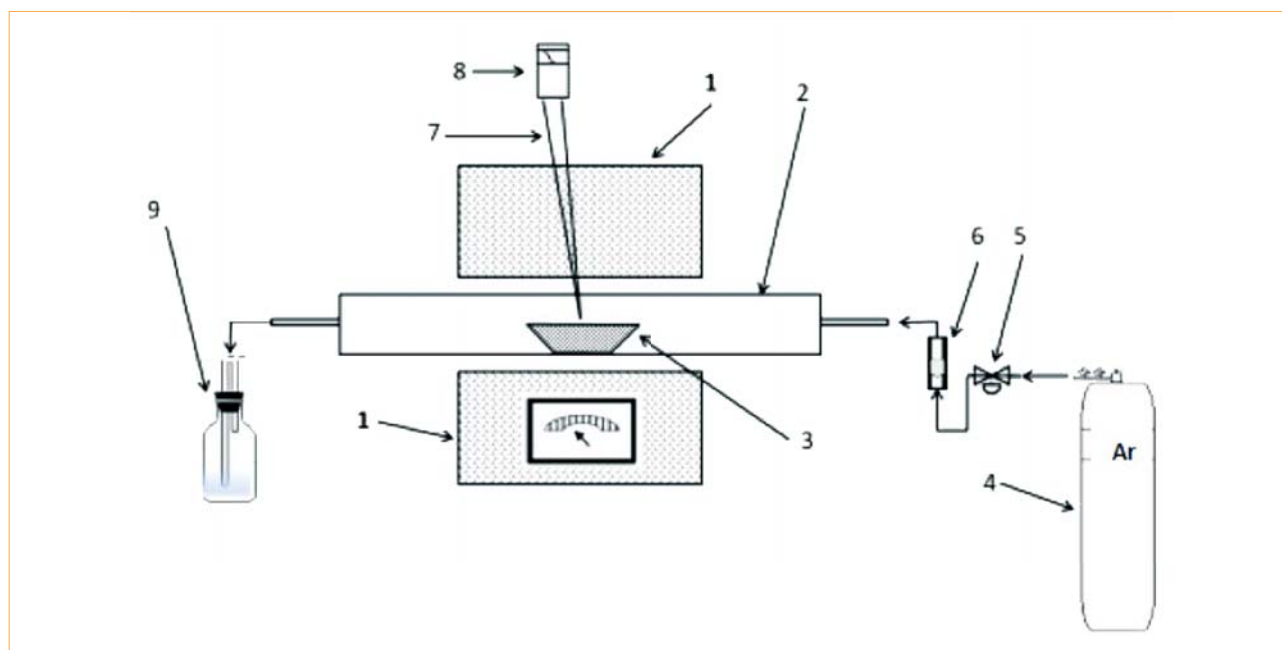


Fig. 1. The scheme of laboratorial unit:

1 – electric furnace Nabertherm 50/250/12 with thermal regulated controller B410; 2 – quartzite reactor; 3 – alundum “boat-form boxes” with hitch; 4 – balloon with argon; 5 – tap; 6 – flowmeter RM-GS 004 KL4; 7 – thermocouple PP-1; 8 – secondary sensor KSP-4; 9 – gas absorption vessel

The experimental technique concludes in the following operations. Non-magnetic fraction of dust, which was obtained after magnetic separation, was mixed with preset amount of ammonium chloride in various mass relations. Consumption of ammonium chloride was varied from 1.1 to 1.7 times (parts of its stoichiometric required amount – SRA), which exceeds SRA for chlorination of lead oxide. The experimental temperature range constituted 300–600 °C.

Amount of initial hitch of non-magnetic dust fraction was permanent for all experiments and made 100 g. Mixture of dust and ammonium chloride was held at preset temperature in inert argon medium, which was introduced in the reaction area of the furnace with rate 100 ml/min. Argon consumption was controlled by a flowmeter RM-GS 004 KL4. Roasting duration was 60 min in all experiments. Then emitted gases were absorbed in the special vessel with lime milk. After termination of the required time, the furnace was switched off and cooled in argon flow, then crucible with contained cinder was evacuated, weighed and subjected to analysis for lead, zinc and iron content.

Each experiment was repeated three times at preset temperature and ammonium chloride consumption in order to obtain reliable results. The average value was considered as the final result.

Results and discussion

Thermodynamic regularities of interaction between lead and zinc oxides and ammonium chloride in the conditions of low-temperature roasting

The model of thermodynamic reaction of chlorination of the components of non-magnetic dust fraction by ammonium chloride is considered from the point of view of established results of phase composition. Evaluation of possible

direction of reactions was carried out via variation of thermodynamic values in the system. Thermodynamic calculations were finalized in calculation of Gibbs free energy (ΔG_T°) for these reactions and in establishment of their relation with the temperature. Such calculations were conducted using the software HSC Chemistry 8.1.5, produced by Outotec Technologies.

Thermodynamic analysis included interaction of the main oxides (lead, zinc, iron) with ammonium chloride within the temperature range 573–973 K. Behaviour of impurity metal oxides (Cu, Ni, Cd etc.), which are presented in non-magnetic dust fraction, was not observed.

Pure solid oxide (MeO_{sol}) was accepted as a standard state of lead and zinc oxides. Pure MeCl_2 gas was accepted for lead and zinc chlorides, and pure solid chloride ($\text{FeCl}_{2\text{sol}}$, $\text{FeCl}_{3\text{sol}}$) – for iron chloride.

The results of thermodynamic calculations are displayed in the **Table 3**.

It can be seen that selective lead chlorination within the temperature range 300–600 °C is more preferable in comparison with selective zinc chlorination (reactions 5, **b**). Thermodynamic possibility of the reactions (5) and (6) is observed just at the temperature 300 °C and it increases with the temperature rise. At the temperature 600 °C, the value of Gibbs free energy in the reaction (5) becomes negative and makes $\Delta G_{600^\circ\text{C}} = -179.43$ kJ/mol.

As for the temperature range from 300 to 600 °C, the values of Gibbs free energy in the reactions (1) and (2) are rather different: the negative values of ΔG_T° for zinc chlorination reaction with forming of solid zinc chloride for the reaction (2) exceed the values of Gibbs free energy for zinc oxide chlorination reaction with forming of gaseous zinc chloride for the reaction (1) more than by 1.5 times.

Table 3. Calculated values of Gibbs free energy for the reactions of lead oxide and zinc chlorination by ammonium chloride

No.	Reactions	Temperature, °C			
		300	400	500	600
		ΔG, kJ/mol			
1	$\text{ZnO} + 2\text{NH}_4\text{Cl} = \text{ZnCl}_{2(\text{gas})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	32.13	-23.17	-77.34	-130.3
2	$\text{ZnO} + 2\text{NH}_4\text{Cl} = \text{ZnCl}_{2(\text{sol.})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	-24.81	-66.21	-107.48	-148.07
3	$\text{ZnFe}_2\text{O}_4(\text{sol.}) + 2\text{NH}_4\text{Cl} = \text{ZnCl}_{2(\text{gas})} + \text{Fe}_2\text{O}_3(\text{sol.}) + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	58.29	4.68	-47.95	-99.61
4	$\text{ZnFe}_2\text{O}_4(\text{sol.}) + 2\text{NH}_4\text{Cl} = \text{ZnCl}_{2(\text{sol.})} + \text{Fe}_2\text{O}_3(\text{sol.}) + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	1.35	-38.36	-78.09	-117.35
5	$\text{PbO} + 2\text{NH}_4\text{Cl} = \text{PbCl}_{2(\text{gas})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	-14.19	-70.52	-125.62	-179.43
6	$\text{PbO} + 2\text{NH}_4\text{Cl} = \text{PbCl}_{2(\text{sol.})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	-97.94	-137.97	-177.21	-218.51
7	$\text{FeO} + 2\text{NH}_4\text{Cl} = \text{FeCl}_{2(\text{gas})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	70.65	14.91	-39.64	-92.94
8	$\text{FeO} + 2\text{NH}_4\text{Cl} = \text{FeCl}_{2(\text{sol.})} + 2\text{NH}_{3(\text{gas})} + \text{H}_2\text{O}_{(\text{gas})}$	-27.31	-66.14	-104.08	-141.05
9	$\text{Fe}_2\text{O}_3 + 6\text{NH}_4\text{Cl} = 2\text{FeCl}_{3(\text{gas})} + 6\text{NH}_{3(\text{gas})} + 3\text{H}_2\text{O}_{(\text{gas})}$	70.65	14.91	-39.64	-92.94
10	$\text{Fe}_2\text{O}_3 + 6\text{NH}_4\text{Cl} = 2\text{FeCl}_{3(\text{sol.})} + 6\text{NH}_{3(\text{gas})} + 3\text{H}_2\text{O}_{(\text{gas})}$	-254.8	-308.7	-362.16	-414.53

It testifies on the fact that favourable conditions for lead chlorination and its sublimation in the form of chloride are created in the conditions of low-temperature roasting of dust non-magnetic fraction by ammonium chloride. In this case zinc chlorides are concentrated in solid phase of a cinder. High possibility of zinc ferrite destruction within the low temperature range 300–600 °C, according to the reactions (3) and (4) with forming of $\text{ZnCl}_{2(\text{gas})}$ and $\text{ZnCl}_{2(\text{sol.})}$ respectively, is considered as rather high. At the same time, thermodynamic possibility of the reaction (4) with forming of solid zinc chloride is more preferable.

As for the high temperature range 500–600 °C, differences of Gibbs free energy values for zinc and lead chlorination reactions, accompanying by forming of their chlorides in gaseous form, become narrow. That's why we could not provide selective lead extraction from dust at high roasting temperatures. Obtained fumes, together with lead chloride, will contain zinc and iron chlorides, what will enlarge in its turn the expenses for their consequent processing.

High thermodynamic possibility of interaction between iron oxides (Fe^{2+} , Fe^{3+}) and ammonium chloride is noted. High negative Gibbs free energy values for the reactions (7) – (10) within the whole examined temperature range show thermodynamic possibility of forming of solid iron chlorides FeCl_2 and FeCl_3 . It can be testified that Fe is not sublimated in the conditions of low-temperature roasting and remains in the form of solid chlorides FeCl_2 and FeCl_3 . Depth of zinc and lead chlorination, as well as their distribution between roasting products (cinder, fumes) is determined not only by temperature variation, but also by consumption of ammonium chloride. To provide maximal selective lead extraction in fumes, it is necessary to establish the optimal technological parameters of roasting (ammonium temperature and its consumption). In order to check the formulated conclusions, laboratorial studies on lead extraction from non-magnetic dust fraction were conducted in dependence on roasting temperature and ammonium chloride consumption.

Laboratorial experiments on low-temperature roasting of non-magnetic dust fraction together with ammonium chloride

Initial hitch, which contains non-magnetic dust fraction in the amount 100 g and various consumption of ammonium chloride, was subjected to roasting at the preset temperature. Ammonium chloride was added in 4 variants based on the following calculation: exceeding its stoichiometric required amount (SRA) for lead oxide chlorination according to the reaction (2) by 1.1, 1.3, 1.5 and 1.7 times. The experiments were carried out at the temperature 300, 400, 500 and 600 °C.

Roasting was conducted at the unit presented in the Fig. 1, according to the above-described technique. Each experiment was accompanied by control of cinder output and its composition for lead, zinc and iron content in it. Amount of fumes was determined via difference between initial hitch and obtained cinder, taking into account lead, zinc and iron content in them. Cinder output made in average 90 % of initial hitch weight.

Influence of temperature on residual lead and zinc content in a cinder is displayed in the Fig. 2.

Ammonium chloride consumption was permanent in the conducted experiments and exceeded its stoichiometric required amount for lead oxide chlorination according to the reaction (2) by 1.5 times. Duration of experiments was 60 min.

Comparative analysis of the results presented in the Fig. 2 shows that temperature rise provides significant effect on decrease of lead content in the final product (cinder). At the same time influence of temperature on zinc content in a cinder is slight. Dramatic lowering of lead content in a cinder is observed within the temperature range 300–500 °C. Minimal lead content in a cinder (0.2 %) was achieved at the temperature 600 °C. Further temperature rise has practically no influence on lowering of lead content in a cinder.

Small increase of zinc content in a cinder from 46.7 % to 47.73 % is observed when the temperature rises from 300 to 600 °C. This established fact correlates well with

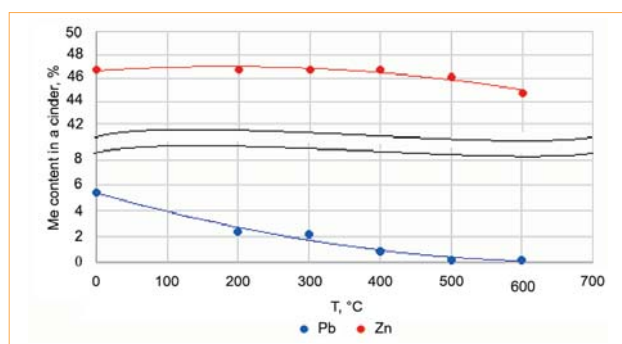


Fig. 2. Relationship between lead and zinc content in a cinder and temperature

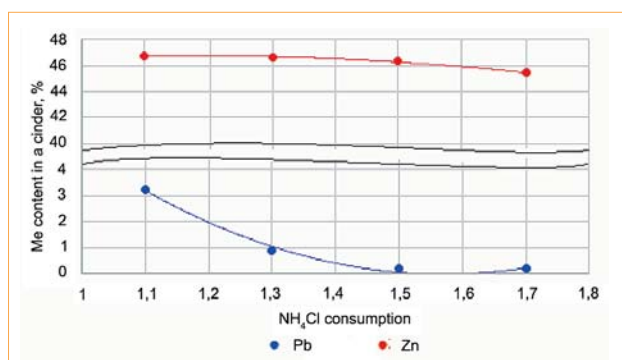


Fig. 4. Relationship between lead and zinc content in a cinder and NH_4Cl consumption

conclusions of thermodynamic analysis, that start of zinc chlorination according to the reaction (1) with its consequent sublimation as chloride can be awaited at increased temperatures, together with lead chlorination according to the reaction (2).

The best results, providing high selective lead extraction in fumes, are achieved at the temperature 500 °C. It can be clearly seen in the Fig. 3, where relationship between variation of lead and zinc content in fumes and temperature is shown; lead and zinc content in fumes are determined via calculation, based on the amount of initial hitch, cinder output and lead and zinc content in them.

Maximal lead content in fumes (equal to 56.17 %) was achieved at the temperature 500 °C. At the same time, zinc content in fumes constituted only 0.02 %. Further temperature rise increases dramatically zinc content in fumes, what leads to decrease of lead content in them.

Influence of ammonium chloride on variation of lead and zinc content in a cinder is shown in the Fig. 4.

Conduction of roasting of non-magnetic dust fraction with NH_4Cl addition provides minimal (up to 0.2 %) lead content in finished cinder. At the same time, general features of the curve displaying relationship between lead and zinc content and NH_4Cl consumption (within the range of consumption varying from 1.1 to 1.7 of SRA) repeats the curve of their relationships of the temperature (see Fig. 4).

It can be seen from the Fig. 4 that increase of NH_4Cl consumption from 1.1 to 1.5 of SRA has no essential effect on zinc content in obtained cinder; at a consumption of NH_4Cl equal to 1.1, the zinc content in the candle is 46.73%,

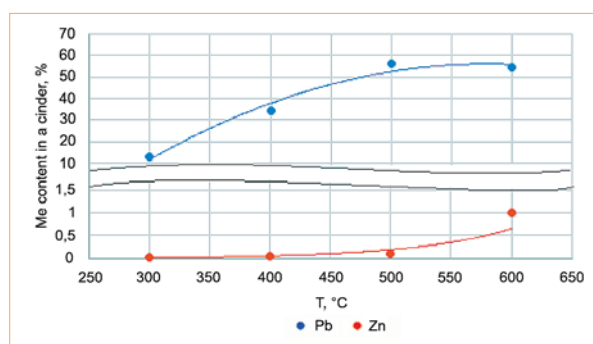


Fig. 3. Relationship between lead and zinc content in fumes and temperature

and at 1.5, a slight decrease in zinc to 46.31 % was found. The minimum zinc content in the cinder equal to 45.43 % was achieved with a consumption of NH_4Cl equal to 1.7 of the SRA.

Dramatic lowering of lead content (from 5.48 % in initial material to 0.2 % in a cinder) is observed within the range of small NH_4Cl consumption values (from 1.0 to 1.5 of SRA); however, further increase of this consumption finalizes in only small its lowering in a cinder (with a SRA of 1.7 – 0.18 % Zn). It was established that optimal NH_4Cl consumption, which provides simultaneous minimal lead value (0.2 %) and maximal zinc content in a cinder (46.31 %), corresponds NH_4Cl consumption exceeding its stoichiometric required amount (SRA) for lead chlorination according to the reaction (2) by 1.5 times.

Based on the conducted experiments, the optimal technological parameters for low-temperature roasting of non-magnetic dust fraction together with ammonium chloride were established: temperature – 500 °C, NH_4Cl consumption – the value exceeding its stoichiometric required amount (SRA) for lead chlorination according to the reaction (2) by 1.5 times, roasting duration – 60 min.

These optimal technological parameters provide obtaining of a cinder with typical foxy-red colour (Fig. 5).

The results of chemical composition of products, which were obtained after magnetic separation and chloridizing roasting of non-magnetic dust fraction at the optimal technological parameters, are presented in the Table 4.

It can be seen that minimal lead content (0.2 %) was achieved in a cinder, which was obtained after roasting of non-magnetic dust fraction together with ammonium



Fig. 5. General view of initial non-magnetic fraction and obtained cinder

Table 4. Chemical composition of processing products


Products	Content, % (mass.)					
	Zn	Pb	Fe	SiO ₂	O	Others
Magnetic fraction	2.99	0.72	67.82	0.37	26.88	1.21
Non-magnetic fraction	46.74	5.48	18.87	7.27	18.89	2.75
Cinder	49.46	0.2	19.97	7.69	14.51	8.17
Lead fumes	0.1	56.17	0.02	–	1.91	0.42

chloride. Large lead content in fumes (56 %) testifies on its high selective extraction during after roasting of non-magnetic dust fraction together with ammonium chloride. The obtained results display high efficiency of the chosen direction of investigations in the field of development of combined technology for processing of dusts from iron and steel works; this technology provides maximum selective extraction of Fe, Zn and Pb from this dust in aimed products.

Conclusions

The method of selective lead extraction from zinc-containing dust, which was preliminary selected via magnetic separation of its non-magnetic fraction, was suggested. This dust was obtained at “Kazferrostal” JSC after processing of iron and steel scrap in electric arc furnaces via low-temperature roasting together with ammonium chloride. The results of thermodynamic calculations show that use of NH₄Cl as chloridizing reactant in the conditions of low-temperature roasting allows to obtain lead fumes in the form of lead chloride with minimal zinc content. It was established that the values of Gibbs free energy for reduction reaction of lead oxide by ammonium chloride within the area of low temperatures 300–600 °C exceeds similar parameters of zinc chlorination by 1.5 times.

Optimal technological parameters, which provide maximal effect in selective lead extraction from dust were established: temperature – 500 °C, NH₄Cl consumption – the value exceeding its stoichiometric required amount (SRA) for lead chlorination by 1.5 times, roasting duration – 60 min. A cinder with the following composition, % (mass.): ~49 Zn; 0.2 Pb; 19 Fe; 8 SiO₂ and other, was obtained. Lead extraction in fumes exceeded ~97 %.

The obtained results will be used for development of a general concept for complex technology for processing of zinc-containing dust and other products with similar composition from iron and steel enterprises. 

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