

Lead and zinc distribution in chloridizing sintering of zinc dust obtained after processing of different kinds of raw materials in the iron and steel industry

N. K. Dosmukhamedov, Professor, Department of Metallurgy and Mineral Processing¹, e-mail: nurdos@bk.ru
I. N. Nursainov, Doctoral Student, Department of Metallurgy and Mineral Processing¹, e-mail: ilyasnursainov@gmail.com
G. M. Koishina*, Associated Professor, Department of Metallurgy and Mineral Processing¹, e-mail: gulzada.koishina@mail.ru
E. E. Zholdasbay, Associated Professor, Head of Department of Mining, Metallurgy and Natural Sciences²
V. A. Kaplan, Academic Consultant, Department of Material and Interfaces³, e-mail: valery.kaplan@weizmann.ac.il

¹ Satbayev University, Almaty, Kazakhstan.

² Zhezkazgan University named after O. A. Baikonurov, Zhezkazgan, Kazakhstan.

³ Weizmann Institute of Science, Rehovot, Israel.

The paper studies the effect of temperature and NH_4Cl consumption on lead extraction from dust of various compositions with determination of optimal sintering parameters. New data on metal distribution between sintering products of dust No. 1 and No. 2 from processing ferrous scrap metal in electric arc furnaces and from smelting iron ore raw materials in an electric steelmaking furnace, respectively, are obtained. Comprehensive analytical studies of intermediate and final products are performed using XRF spectroscopy, XRD analysis and electron microscopy. This ensures quality control of the obtained products during the implementation of the technological process of dust sintering together with ammonium chloride. Based on the experiments, a high distribution of Pb from dust No. 1 and No. 2 into sublimates was achieved – 98% and 97.6%, respectively, with its residual content in clinker – 2.0% and 2.4%. At the same time, low zinc extraction from dust No. 1 and No. 2 into sublimates is shown – 0.04% and 0.06%, respectively. The technology proposed in the work ensures environmental safety of the dust processing and the production of valuable technogenic raw materials – lead sublimates and zinc-containing clinker, which can be used as additional raw materials for the production of commercial lead and zinc.

Key words: dust, sintering, lead, zinc, ammonium chloride, distribution, mathematical model.

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Introduction

Recycling of production waste not only allows for more rational use of natural resources, which leads to increased production efficiency, but also improves the environmental situation both at the enterprise itself and in its region. In the steelmaking industry, man-made waste reaches 30% of the total steel production volume, including about 80% of slag and about 20% of gas cleaning dust and sludge [1, 2]. About 10-15 million tons of this waste are generated annually in the world. It is known that 1-2% of raw materials entering the smelting unit turn into dust, while for some technologies this figure can reach 10% [2–4]. Accumulating waste from electric arc steelmaking furnaces creates pollution zones, having a negative impact on the atmosphere and soils, which, in turn, affects human health and ecosystems [5].

Current dust recycling technologies are generally inefficient or economically impractical and can be divided into three categories.

The first category includes technologies that do not extract zinc from the dust, such as agglomeration with return to the blast furnace [5, 6] and stabilization with a slag or cement layer [7, 8]. Although these methods are low-cost, agglomeration does not allow zinc to be removed, making it acceptable only for low concentrations of this metal. Stabilization also does not solve the problem of heavy metal leaching.

The second category includes pyrometallurgical zinc extraction methods such as Waelz, FASTMET and OXYCUP technologies, which focus on high-temperature reduction of oxides. The main disadvantage of these methods is the high consumption of reducing agents and corrosion of equipment [9–11].

The third category represents hydrometallurgical methods using acid and alkaline leaching. Acid methods provide a high yield of zinc, but they lead to the concentration of a significant amount of impurities in the final product [12, 13].

Alkaline methods require high temperatures and alkali concentrations, which limits their use [14–16]. As a result, many enterprises simply accumulate and store dust,

*Correspondence author.

which causes environmental problems, including large volumes of waste in landfills.

Despite the existing shortcomings, research and development in the field of dust processing continues. One promising direction is the combination of different methods, for example, dust pre-treatment followed by hydrometallurgical zinc extraction. This approach reduces the content of impurities and increases the efficiency of the process.

Another important area is the development of cleaner and more cost-effective reducing agents for pyrometallurgical processes. Research into the use of biomass and other renewable energy sources can significantly reduce costs and make them sustainable.

Ultimately, the choice of the optimal dust recycling method depends on the specific characteristics of the waste, economic factors and environmental requirements. An integrated approach that takes into account all aspects of the problem is necessary to achieve a sustainable and environmentally friendly solution.

The development of efficient technologies for processing steelmaking waste is becoming a pressing issue for metallurgy, and research in this area is extremely important.

One of the wastes at KazFerroSteel LLP (Almaty) is dust rich in metals, in particular zinc and lead, which is a serious technological and environmental problem.

The aim of this study is to investigate the distribution of zinc and lead during chlorination sintering of dust in order to identify the possibilities of its utilization.

Studying the distribution of zinc and lead in dust processing products will allow to develop an optimal technological scheme for its utilization. Possible options include extracting valuable metals for reuse in industry or processing into environmentally friendly materials suitable for the construction industry or road construction.

An analysis of current dust handling methods at KazFerroSteel LLP revealed the need to optimize processes in order to reduce the negative impact on the environment. In particular, the existing dust collection systems and the possibilities of their modernization to improve the efficiency of dust collection and separation by fractions and composition were considered. The obtained results formed the core of the new chlorination technology for processing zinc dust at KazFerroSteel LLP [17, 18].

It is expected that the results of the present research will allow to propose specific technological solutions for processing dust enriched with zinc and lead, taking into account economic and environmental factors. This, in turn, will contribute to improving the environmental situation in Almaty and increasing the efficiency of resource use at the enterprise.

Research methods and experimental procedure

Two types of dust were subjected to sintering: dust No. 1 – non-magnetic fraction of dust from smelting of ferrous scrap in an electric arc furnace [18] and dust No. 2 – from smelting of iron ore raw materials in an electric steelmaking furnace. The chemical compositions of the dust are shown in **Table 1**.

The results of the qualitative characteristics of dust No. 2, obtained by XRF spectroscopy (**Fig. 1**) show the presence of Zn, Pb, Fe and a wide range of associated impurities.

The results of the XRD analysis of the metal speciation showed an identical phase composition to that of dust No. 1, previously established in [18]. Lead and associated elements in dust No. 2 are mainly present in the form of their oxides. Zinc, in contrast to its speciation in dust No. 1, along with zincite (ZnO), is present in large quantities, up to 26%, in the form of ferrite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$). Iron was found in the form of wustite (FeO) and hematite (Fe_2O_3) (**Fig. 2**).

Unlike dust No. 1, dust No. 2 is characterized by a low content of lead and magnetite in its chemical and phase composition (**Fig. 2**). This indicates that for this category of dust, preliminary magnetic separation is not necessary.

The experimental procedure was as follows. The dust sintering experiments were conducted at different temperatures of 400, 500, and 600 °C. The duration of the experiments was

Table 1
Chemical composition of the original dust

Original Sample	Content, % wt.					
	Zn	Pb	Fe	Si	O	Other
Dust No. 1	46.74	5.48	18.87	7.27	18.89	2.75
Dust No. 2	37.69	1.36	22.48	9.42	20.1	8.95

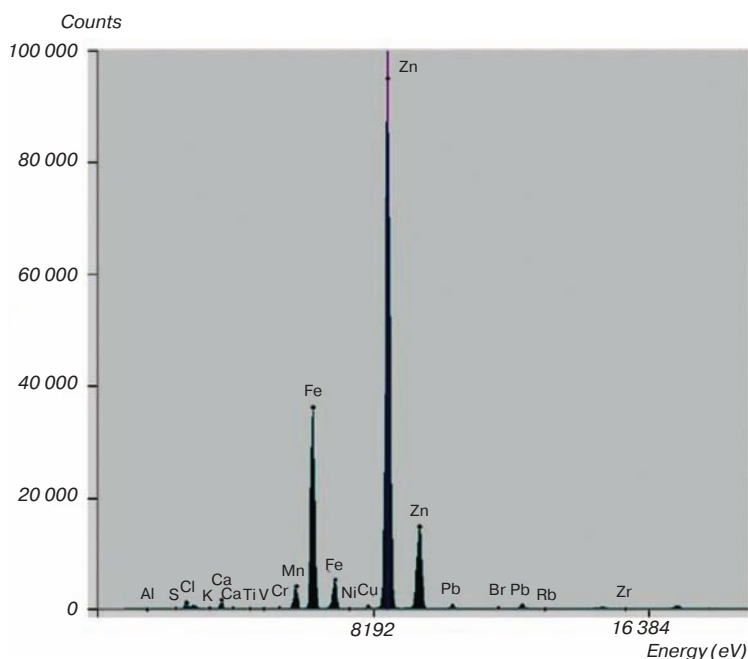


Fig. 1. XRF spectroscopy of dust No. 2

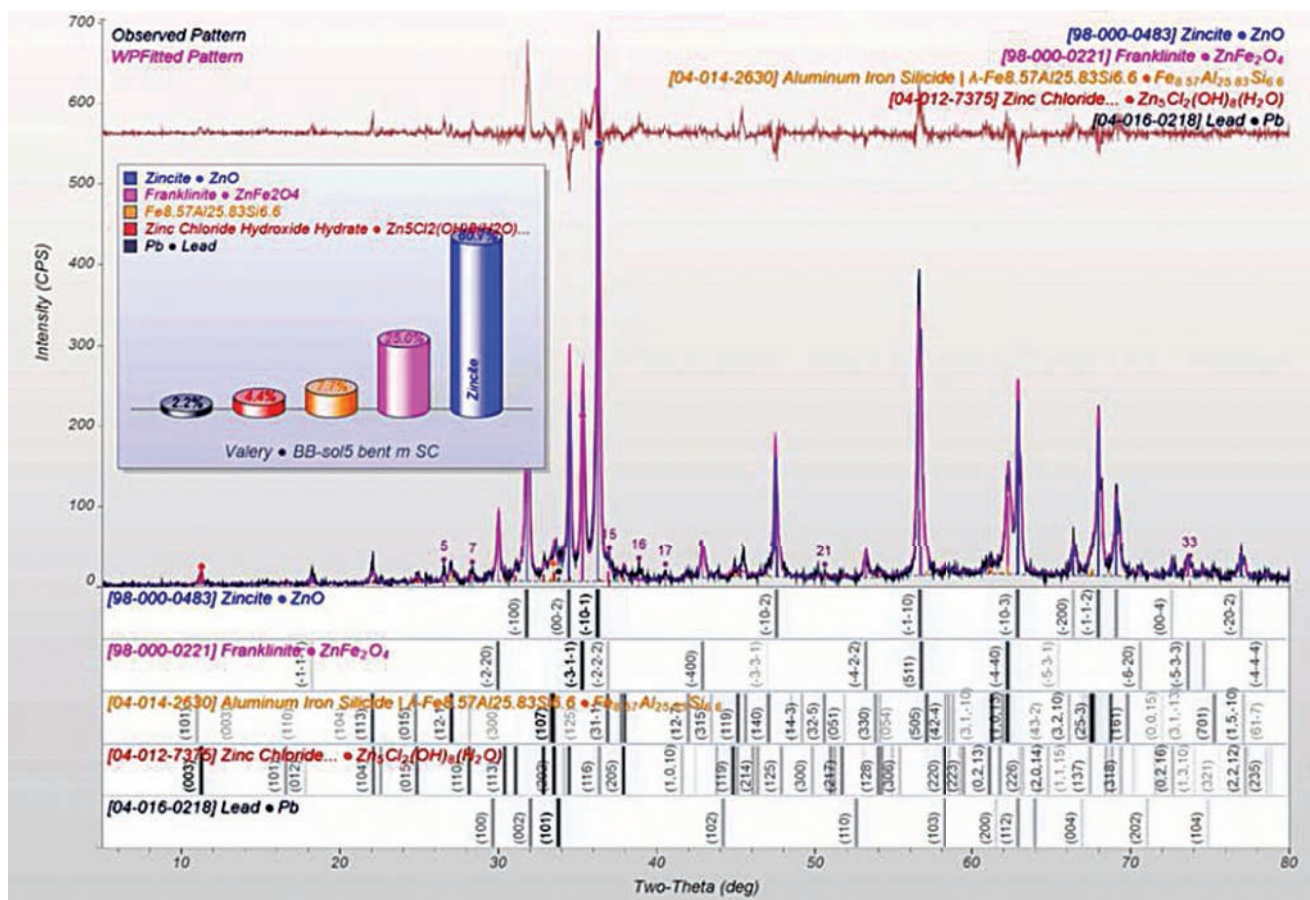


Fig. 2. Results of XRD analysis of dust No. 2

constant and amounted to 60 minutes. In all experiments, a given amount of NH_4Cl was added to the initial dust sample (100 g) at the rate of: 1.1; 1.3; 1.5 (fractions of the SRA unit) times exceeding its consumption from the stoichiometric required amount (SRA) for the reduction of lead oxide. The same conditions for dust sintering experiments No. 1 and No. 2 made it possible to evaluate the use of the technology for processing the dust of different types and compositions with a comparative analysis of the results on the distribution of metals between the sintering products. The setup described in detail in [17] was used to conduct the experiments. After each experiment, the quantity and elemental composition of the obtained clinker were monitored. Each experiment was conducted three times under the same conditions.

Results and discussion

During the experiments, identical patterns in the behavior of metals during sintering were established for both dust No. 1 and dust No. 2, as in laboratory conditions.

During sintering of dust together with ammonium chloride, selective sublimation of lead in the form of its chloride was established in the entire studied temperature range. This is evidenced by the low residual lead content in the clinker obtained after the experiments.

Extraction of Pb into sublimates, %

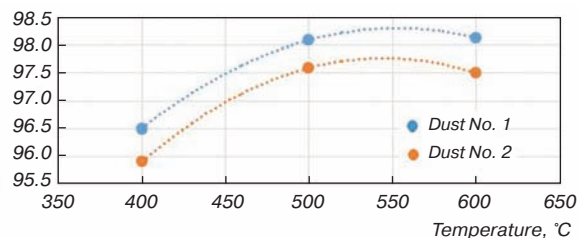


Fig. 3. Lead extraction dependence on temperature: NH_4Cl consumption = 1.5; τ = 60 minutes

Comparative results on the effect of temperature on the extraction of lead into sublimates from dust No. 1 and dust No. 2 are shown in Fig. 3.

Fig. 3 shows that the curves of lead extraction from both dust No. 1 and dust No. 2, depending on the temperature increase, show an identical character, regardless of the change in the initial composition of the dust. In the temperature range of 400–500 °C, a sharp increase in lead extraction is established. An increase in temperature (>500 °C) has virtually no effect on the increase in extraction: for dust No. 2 it is absent at all, and for dust No. 1 the increase is minimal (Fig. 3).

The higher the lead content in the initial dust, the higher the extraction of lead from dust. This statement

is in good agreement with the theory of the process and the results of laboratory experiments obtained in [17]. At low process temperatures, ammonium chloride actively interacts with lead oxide to form gaseous lead chloride and solid zinc chloride. This is evidenced by the results of micromorphology of the clinker obtained after the experiments, in which only solid forms of zinc and iron chlorides were found. At high temperatures ($>400\text{ }^{\circ}\text{C}$), ammonium chloride completely decomposes, which leads to a decrease in its reactivity. To maintain an intensive reaction of PbO reduction to PbCl_2 , it is necessary to increase the consumption of NH_4Cl , which does not seem effective from the point of view of material costs for the technology. In addition, with an increase in NH_4Cl consumption and temperature, the probability of sublimation of zinc and iron chlorides increases [17]. This will significantly reduce the quality of the resulting lead sublimates and increase material costs during further processing of the sublimates.

The choice of the flow rate of NH_4Cl specified in the experiments only based on the required amount from the lead content in the dust shows that its amount will not be sufficient to recover zinc ferrite. Indeed, the results of the XRD analysis of the clinker obtained with the optimal sintering parameters of dust No. 2 showed the presence of zinc ferrite in it (Fig. 4). At the same time, lead was

not detected in the clinker, which indicates its complete removal from the dust into sublimates.

The presence of zinc ferrite in the clinker does not seem critical, since with further high-temperature sintering of the clinker together with calcium chloride, its complete destruction is achieved with the maximum transfer of zinc into sublimates in the form of its chloride.

The obtained results show that the optimum process temperature is $500\text{ }^{\circ}\text{C}$, at which high extraction of lead from dust No. 1 and dust No. 2 is achieved: 97.7% and 97.6%, respectively. Based on the above, it follows that the study of the effect of ammonium chloride consumption on lead extraction from dust is of great interest. Fig. 5 shows the dependence of lead extraction from dust No. 1 and dust No. 2 on NH_4Cl consumption, obtained at a constant process temperature of $t = 500\text{ }^{\circ}\text{C}$.

The established regularity fully confirms the above-stated arguments on the effect of ammonium chloride consumption on lead extraction from dust. It is evident that lead extraction into sublimates increases with an increase in ammonium chloride consumption.

The obtained results show the fundamental possibility of using the developed technology for selective extraction of lead from dust of various compositions with obtaining high-quality lead sublimates at optimal

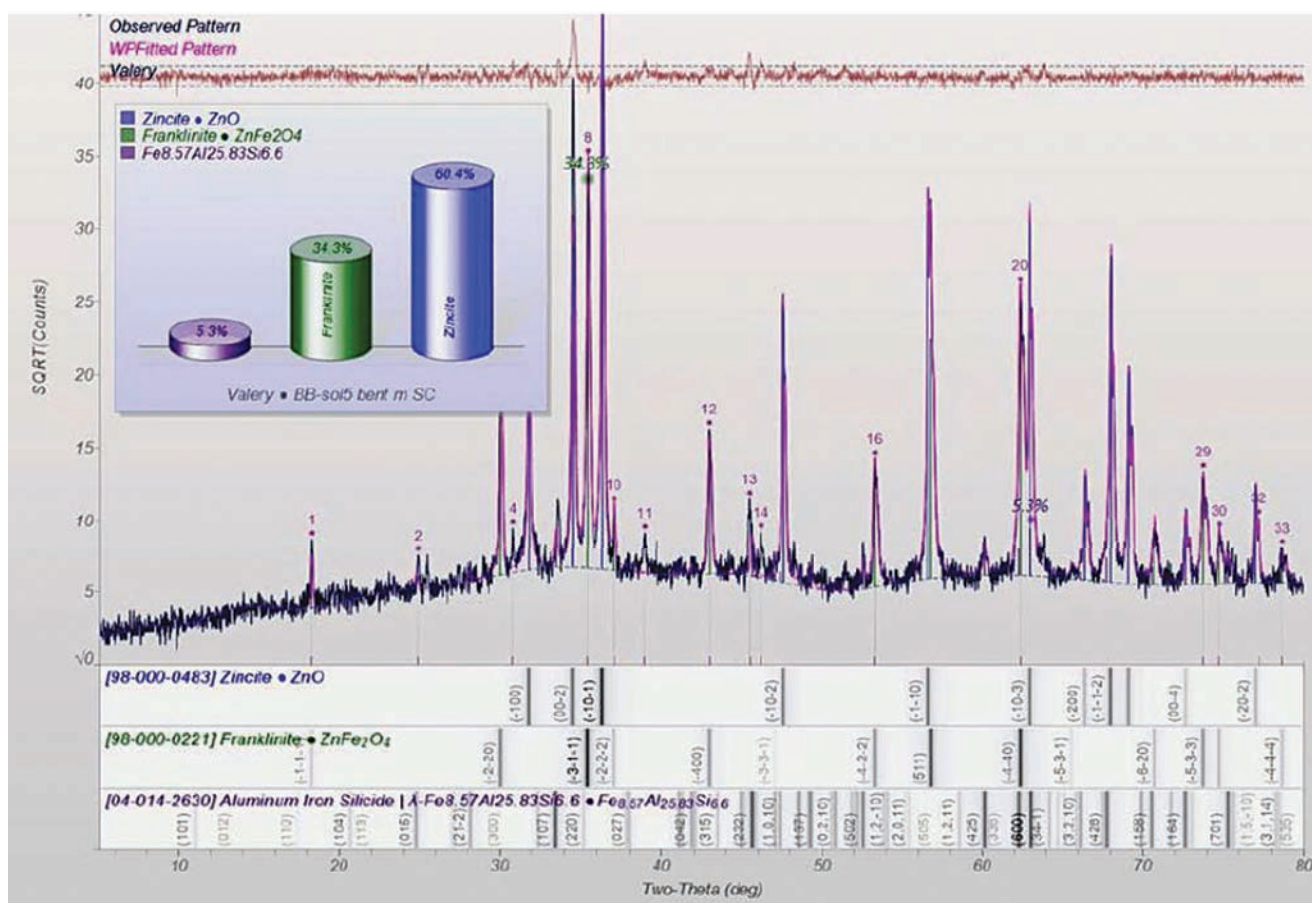


Fig. 4. Results of XRD analysis of clinker from sintering dust No. 2: $t = 500\text{ }^{\circ}\text{C}$, $\tau = 60\text{ min}$, NH_4Cl consumption = 1.5 SRA-based

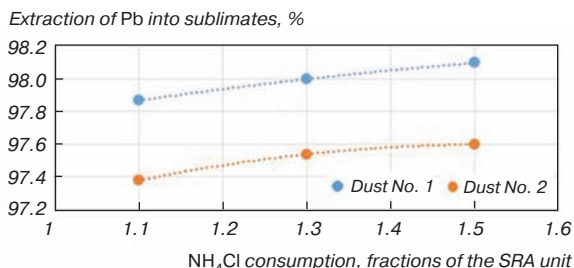


Fig. 5. Dependence of lead extraction into sublimates on NH₄Cl consumption: *t* = 500 °C, τ = 60 minutes

sintering parameters: *t* = 500 °C, τ = 60 min, NH₄Cl consumption – 1.5 times exceeding its SRA-based consumption for lead recovery. Increasing the ammonium chloride consumption above the optimal level, although it leads to insignificant lead extraction, however, from an economic point of view, does not seem expedient.

Based on the results on the quantity and composition of the initial dust and the obtained products, averaged material balances of sintering for dust No. 1 and dust No. 2 together with ammonium chloride were calculated for optimal process conditions.

Fig. 6 shows the results of a comparative analysis of the distribution of lead and zinc between the sintering

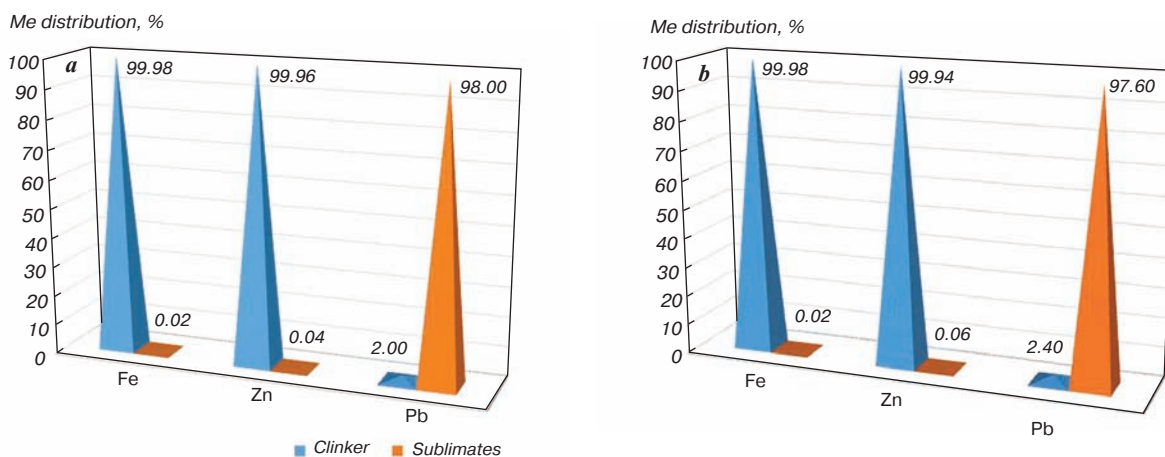


Fig. 6. Distribution of lead and zinc between the products of sintering dust No. 1 (a) and dust No. 2 (b)

Table 2
Sample data array accepted for mathematical processing

Chemical composition of the original dust, wt. %			Parameters			Extraction of Me into sublimates, %		Extraction of Me into clinker, %	
Zn	Pb	Fe	NH ₄ Cl consumption	<i>t</i> , °C	τ , min	Zn	Pb	Zn	Pb
Dust No. 1									
46.74	5.48	18.87	1.1	400	60	0.02	95.79	99.98	4.21
46.74	5.48	18.87	1.3	400	60	0.01	95.90	99.99	4.1
46.74	5.48	18.87	1.5	400	60	0.02	96.10	99.98	3.9
46.74	5.48	18.87	1.1	500	60	0.02	97.71	99.98	2.29
46.74	5.48	18.87	1.3	500	60	0.03	97.79	99.97	2.21
46.74	5.48	18.87	1.5	500	60	0.04	98.00	99.96	2
46.74	5.48	18.87	1.1	600	60	0.32	98.41	99.68	1.59
46.74	5.48	18.87	1.3	600	60	0.35	98.51	99.65	1.49
46.74	5.48	18.87	1.5	600	60	0.41	98.68	99.59	1.32
Dust No. 2									
37.69	1.36	22.48	1.1	400	60	0.05	95.66	99.95	4.34
37.69	1.36	22.48	1.3	400	60	0.04	95.81	99.96	4.19
37.69	1.36	22.48	1.5	400	60	0.05	95.90	99.95	4.1
37.69	1.36	22.48	1.1	500	60	0.04	97.22	99.96	2.78
37.69	1.36	22.48	1.3	500	60	0.05	97.41	99.95	2.59
37.69	1.36	22.48	1.5	500	60	0.06	97.60	99.94	2.4
37.69	1.36	22.48	1.1	600	60	0.28	98.14	99.72	1.86
37.69	1.36	22.48	1.3	600	60	0.31	98.28	99.69	1.72
37.69	1.36	22.48	1.5	600	60	0.37	98.40	99.63	1.6

products of dust No. 1 and dust No. 2, obtained from the data of the material balances.

The values of lead and zinc distribution between sintering products established as a result of experiments show high results in selective extraction of lead into sublimates, regardless of changes in the composition of the initial dust.

Based on the large array of data obtained as a result of the experimental studies, mathematical processing was carried out. Mathematical models were constructed that adequately describe the extraction of lead into sublimates depending on various parameters.

An array of 57 points representing a set of independent parameters was accepted for mathematical processing. The sample data array is shown in **Table 2**.

As a result of mathematical data processing, a multiple correlation equation was obtained that adequately predicts lead extraction (ε_{pb}) depending on the dust composition:

$$\varepsilon_{pb} = 95.59 + 0.009 \cdot \text{Zn} + 0.17 \cdot \text{Pb}, \quad r = 0.98, \quad (1)$$

where: Zn, Pb – zinc and lead content in dust, wt.%

The multiple correlation equation describing lead extraction from dust as a function of lead content in dust and NH_4Cl consumption is:

$$\varepsilon_{pb} = 96.39 + 0.15 \cdot \text{Pb} + 0.58 \cdot \text{NH}_4\text{Cl}, \quad r = 0.96, \quad (2)$$

where: Pb – lead content in dust, wt.%; NH_4Cl – consumption of stoichiometric required amount (SRA) of ammonium chloride for reduction of lead oxide to chloride, fractions of SRA unit.

The obtained equations (1) and (2) demonstrate a high degree of correlation between the predicted lead extraction and the studied factors. The value of the multiple correlation coefficient (r), close to 1 in both cases, indicates a close relationship between the metal content in the dust and the consumption of ammonium chloride with the efficiency of lead extraction.

Equation (1) highlights the influence of zinc and lead content in dust on lead recovery. The positive coefficients of the Zn and Pb variables indicate that increasing the concentration of these metals contributes to an increase in lead recovery. This may be due to synergistic effects arising from the interaction of metals during processing.

Equation (2) shows the effect of lead content in dust and ammonium chloride consumption. Positive coefficients for the Pb and NH_4Cl variables also indicate that increasing lead content and ammonium chloride consumption favorably influences lead recovery. This confirms the role of ammonium chloride in converting lead oxide into a soluble form, facilitating its recovery.

Comparison of equations (1) and (2) allows to identify key factors influencing lead extraction from dust and to optimize the process for maximum efficiency.

Conclusion

New data on the distribution of metals between the sintering products of dust No. 1 and No. 2 were obtained. The distribution of Pb into sublimates is 98% and 97.6%, respectively; into clinker – 2.0% and 2.4%. The distribution of Zn into clinker is 99.96% and 99.94%, respectively; into sublimates – 0.04% and 0.06%. Almost identical values for the distribution of Pb, Zn between the sintering products of dust No. 1 and No. 2 confirm the stability of the physicochemical patterns of lead reduction occurring during sintering of dust of different compositions and are completely consistent with the conclusions on the selective extraction of lead into sublimates from dust due to the reduction of lead oxide with ammonium chloride.

The developed new quantitative mathematical models, which adequately predict the extraction of lead into sublimates depending on the composition of the initial dust and the NH_4Cl consumption, will be used in further studies on the extraction of zinc from different clinker compositions under conditions of its high-temperature sintering together with CaCl_2 .

Positive results have been recorded in the process of extracting lead by chlorinating sintering. However, the effectiveness of the entire process is directly related to the efficiency of condensation of these sublimates. It is important to establish how reliably the resulting lead vapors and gaseous compounds can be captured.

A thorough analysis of the complete composition of the sublimates is necessary to form a comprehensive picture. Information on all components, including undesirable impurities and accompanying elements, is crucial for selecting the most appropriate technology for subsequent processing. In laboratory conditions, the solution of the task was not possible due to safety requirements. The capture of sublimates and vapors was carried out in an absorption vessel. The content of lead, iron and zinc, as indicated above, was determined by the difference based on the amount and content of metals in the original dust and the resulting clinker.

An important issue is the possibility of using standard technologies to transform the obtained sublimations into products ready for sale. In the event of insufficient efficiency of existing methods or their inconsistency with the specific composition of sublimates, it will be necessary to develop an innovative, specialized process. This task may require additional research and development work, which should be taken into account when assessing the economic profitability of the technology as a whole.

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