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Metallurgy

Enlarged laboratory experiments of ferrous metallurgy dust sintering with calcium chloride

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ABSTRACT
In this work, large-scale laboratory experiments were carried out on high-temperature sintering of clinker with $CaCl_2$. New data were obtained on extracting zinc, lead and related impurities into the targeted products of high-temperature sintering under process scaling conditions. The optimal parameters for high-temperature sintering have been established: t =900 °C, τ = 60 minutes, $CaCl_2$ consumption is 1.3 times higher than its consumption from the stoichiometric required amount (SRA). High-quality zinc sublimates were obtained. The maximum extraction of zinc into sublimates was achieved - 98.2%. New data on changes in the quantitative ratios of clinker ingredients under sintering conditions have been obtained. It has been established that zinc in the form of chloride is concentrated in sublimations. The obtained results form the basis of the final stage of the chlorination technology for processing lead-containing zinc dust from ferrous metallurgy and were used in the developing of a general technology for the selective extraction of zinc and lead into marketable products.

Keywords: dust, lead, zinc, extraction, chlorinating sintering.

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Introduction

Today, accumulated dust from ferrous metallurgy occupies huge areas and requires disposal. Ferrous metallurgy specks of dust, in particular zinc-containing specks of dust, are characterized by a complex chemical and phase composition. The need for their processing lies in the fact that they contain valuable metals. On average, the zinc content in dust is estimated at 8-10%. The return of zinc-containing dust to smelting negatively affects the technological parameters and smelting modes: an increase in the zinc content in the resulting smelting products and the formation of localized deposits in the furnace. Several methods are known, but they are not applicable due to the multi-component composition of the dust obtained under different melting conditions and parameters [[1], [2], [3], [4], [5], [6], [7]]. Processing of zinc sublimates (in the form of oxides) by fuming and other processes leads to environmental degradation, an example is the processing of zinc sublimates at Kazakhmys LLP [[8], [9], [10], [11], [12], [13], [14], [15], [16], [17], [18], [19]]. The use of a pulverized coal mixture leads to a large yield of CO_2.

Based on the above, it follows that solving the problem of complex processing of ferrous and non-ferrous metallurgy dust is an urgent task and requires finding new ways to process them.

In previously completed work [20], we presented the general concept of combined chlorination technology for the complex processing of dust from Kazferrostal LLP (Almaty), obtained from processing ferrous scrap into EAF. The technology includes the selective extraction of iron, lead and zinc into targeted products through stage-by-stage processing. The results of the studies showed the fundamental possibility of developing a technology for producing commercial products from dust: an iron-containing product, high-quality lead and zinc sublimates with a high recovery of lead and zinc in them, over 98%.

At the initial stage of the technology, a magnetic fraction (~70% Fe) is separated from dust using the magnetic separation method, which is sent for agglomeration before blast furnace remelting. The non-magnetic dust fraction (46.74% Zn, 5.48% Pb) is subjected to low-temperature chlorinating sintering with NH_4Cl with selective extraction of lead into sublimates. The resulting zinc-rich clinker, in the second stage, is subjected to high-temperature sintering with $CaCl_2$ with selective extraction of zinc from it into sublimates and the production of final clinker with a high iron content, suitable for producing steel.

Lead and zinc sublimates of high quality can be used as commercial products and sent for sale, or, after processing by known methods (smelting, electrolysis), they can be processed to obtain pure lead and zinc. The minimum content of impurities in them will allow further operations to be carried out with low material costs.

The purpose of this research is to test the reproducibility and accuracy of the results of the method for extracting zinc from clinker by high-temperature sintering with calcium chloride [21] under scaling conditions with the establishment of optimal technological process parameters.

Materials and research methods

As the initial object, clinker was used, obtained after low-temperature sintering of dust with NH4Cl, composition, wt.%: 49.74 Zn; 0.23 Pb; 20.08 Fe; 7.74 SiO2; 18 O and others.

The conditions, experimental procedures and methods for studying the initial and resulting products were similar to those used in laboratory experiments [21]. Technological experiments were carried out with an initial sample of clinker of 500 g. The consumption of $CaCl_2$ in the experiments was 1.0; 1.1; 1.3 and 1.5 times higher than its stoichiometric required amount (SRA) for chlorination of zinc oxide, which was calculated based on the zinc content in clinker. Experiment temperatures were 600, 700, 800, 900 and 1000 °C. Duration – 60 minutes. Each experiment at a given temperature and $CaCl_2$ flow rate was repeated three times in order to be reproducible and obtain accurate results. The average value was taken as the final result.

The number of sublimations was determined by the difference between the initial sample and the resulting clinker, taking into account the content of lead, zinc and iron in them. The degree of extraction of zinc and lead into sublimates was determined by the formula:

$$\mathcal{E}\mathrm{Me} = \frac{(g1\times m1) - (g2\times m2)}{(g1\times m1)}, \qquad (1)$$

where: g_1 – a mass fraction of the element in the original clinker;

m_1 – mass of the initial clinker sample, 500 g;

g_2 - a mass fraction of the element in the clinker after sintering;

m_2 - a mass of clinker after sintering.

Based on the results of experiments, the material balance and extraction of zinc and lead into sintering products were calculated with a comparative analysis of the data.

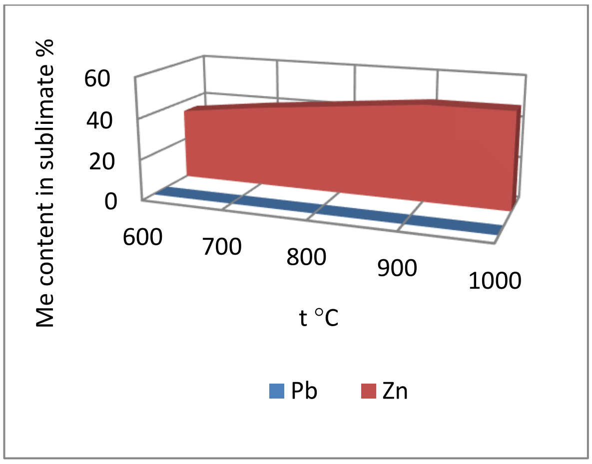
Results and discussion

The established values of Δ H>0 kJ/mol for the reaction of zinc oxide with $CaCl_2$ show that the chlorination process is endothermic and the efficiency of the ongoing reactions during high-temperature sintering of clinker is closely related to temperature.

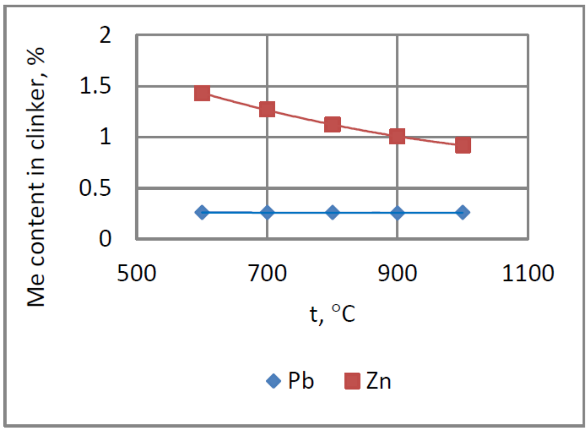
To check the effectiveness of extraction of Zn and Pb from clinker, the mixture (clinker + given $CaCl_2$ flow rate) in an amount of 500 g was subjected to sintering at a temperature of 600-1000 °C for 60 minutes at an airflow rate of 100 ml/min.

The study of the influence of temperature on the dynamics of changes in the content of Zn and Pb in the sintering products was carried out at a constant sintering time of 60 minutes and consumption of $CaCl_2$ 1.3 times higher than its stoichiometric required amount (SRA) for the reduction of zinc compounds to chloride. The choice of parameters is determined by the need to conduct a comparative assessment of the results for reproducibility and accuracy with laboratory experiments.

The effect of temperature on the content of zinc and lead in sublimates and clinker is shown in Fig. 1.



a)



b)

Fig. 1 - Dependence of Zn and Pb content in sublimates (a) and clinker (b) on temperature

As can be seen in Fig. 1 (a), under high-temperature sintering conditions, the main contribution to the formation of sublimates is made by zinc chloride. The Zn content in sublimates changes greatly with increasing temperature: an increase in temperature from 600 to 1000 °C increases the zinc content in sublimates from ~35 to ~41%. At high temperatures of 800 °C and above, the lead content in sublimates is minimal and varies slightly within low limits from 0.003 to 0.002%. The fact seems quite natural, since in the considered temperature range 600-1000 °C, the Zn content in clinker shows a sharp decrease from 1.43 to 0.92%. At the same time, as can be seen in Fig. 1 (b), the nature of the curve of the change in lead content practically does not change and shows a stable value at the level of 0.26%.

SEM images of clinkers obtained after chlorination sintering at a temperature of 700, 800, 900 and 1000 °C are presented in Fig. 2.

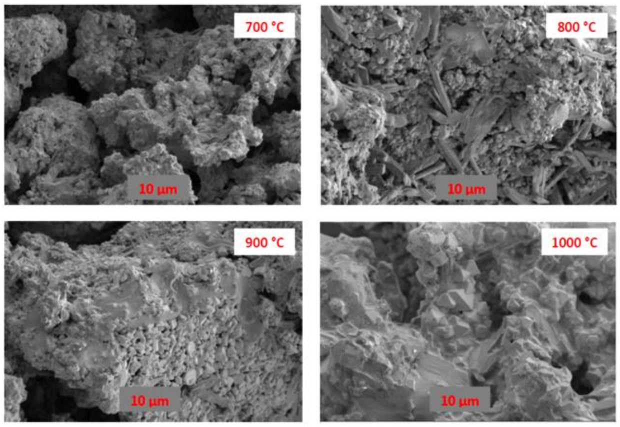


Fig.2 - SEM images of samples (clinker) chlorinated at different temperatures: 700 °C, 800 °C, 900 °C, 1000 °C

In the range of 700-900 °C, with increasing temperature, calcined clinker gradually begins to sinter and undergoes phase transitions. At elevated temperatures due to a decrease in the porosity of the material, kinetic conditions chlorination reactions are hindered: $CaCl_2$ particles are not completely in contact with zinc and lead compounds. At temperatures above 1000 °C, clinker begins to melt (Fig. 2), and at higher temperatures (above 1200 °C) it passes into the liquid phase, which seriously reduces the extraction of zinc and lead into sublimates. Consequently, an increase in the sintering temperature, although it provides high productivity, significantly increases the material costs of the process: the consumption of chlorinating agent increases ($CaCl_2$), and the quality of the resulting sublimates decreases. Based on the obtained results and taking into account the established patterns of the influence of temperature on the quality of the resulting sublimates, as well as taking into account the cost of calcium chloride and energy consumption, a temperature of 900 °C was taken as the optimal temperature for the sintering process.

In the process of high-temperature chlorinating sintering of clinker, the distribution of Zn and Pb between products is significantly affected by the consumption of $CaCl_2$ - as an active chlorinating reagent, providing effective selective chlorination of zinc with low energy consumption and minimal environmental pollution.

The physicochemical essence of the process of high-temperature sintering of clinker with $CaCl_2$ can be interpreted as a direct and indirect mechanism of metal chlorination [22].

The mechanism of direct chlorination can be represented by the reaction:

$$CaCl_2 + MeO = 2CaO + MeCl_2, \quad (2)$$

where solid calcium chloride reacts directly with the metal oxide to form the corresponding chloride.

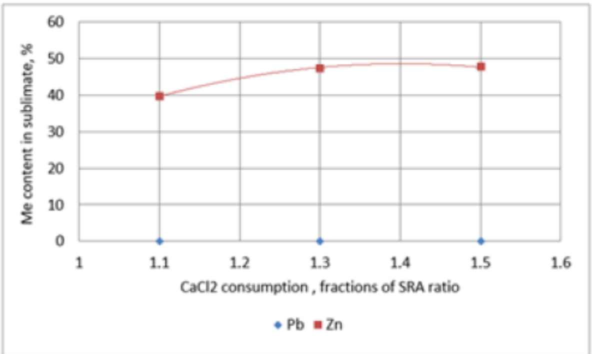
The indirect mechanism of metal chlorination is usually represented as a stepwise one: first, the decomposition reaction of solid $CaCl_2(s)$ occurs due to oxygen to its oxide with the formation of gaseous chlorine ($Cl_2(g)$). The chlorine then reacts with the metal oxide and reduces it to the corresponding chloride. The general mechanism is described by the occurrence of reactions:

$$2CaCl_{2(s)} + O_{2(g)} = 2CaO + 2Cl_{2(g)}, \quad (3)$$

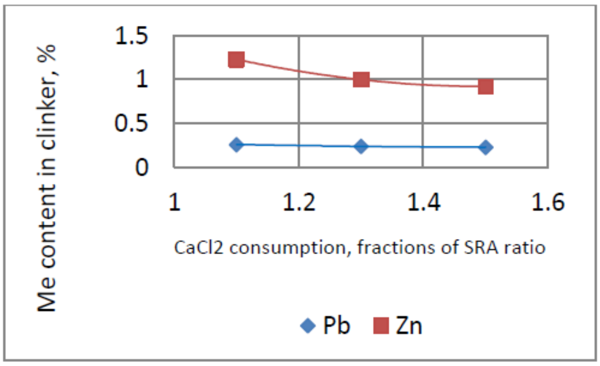
$$2MeO_{(s)} + 2Cl_{2\,(g)} = 2MeCl_{2(g)} + O_{2(g)}. \quad (4)$$

The physicochemical patterns that occur during the sintering of clinker with $CaCl_2$ can be interpreted by the mechanism of direct chlorination, wherein the ZnO-Fe_2O_3-$CaCl_2$ system, at temperatures above the melting point of $CaCl_2$ (>700 °C), calcium chloride with zinc oxide reacts directly according to reaction (1) to form gaseous $ZnCl_2$.

The effect of $CaCl_2$ consumption on the zinc content in sintering products, set at an optimal temperature of 900 °C, is shown in Fig. 3.



a)



b)

Fig.3 - Dynamics of changes in the content of Zn and Pb in sublimates (a) and clinker (b) depending on the consumption of $CaCl_2$: t =900 °C, τ =60 minutes.

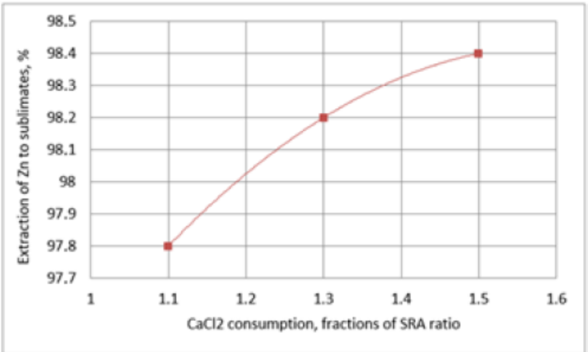
As can be seen in Fig. 3, the Zn content in the sintering products strongly depends on the $CaCl_2$ consumption. Interesting results were obtained in experiments conducted without the addition of $CaCl_2$. When simply heating the clinker at 900 °C for 60 minutes, the residual content of Zn and Pb in the resulting clinker, although insignificant, showed a decrease from the initial 50.46% and 0.24% to 49.78% and 0.21%, respectively. This phenomenon is explained by the fact that at high temperatures, zinc and lead compounds present in clinker in the form of solid chlorides, having high vapor pressure, partially begin to sublime.

With an increase in the consumption of $CaCl_2$ from 1.1 to 1.3 times higher than its consumption from the SRA, the zinc content in the sublimates increases from ~40 to ~50% (Fig. 3 (a)). With a maximum zinc content in sublimates, the lead content in them is minimal and amounts to ~0.002%.

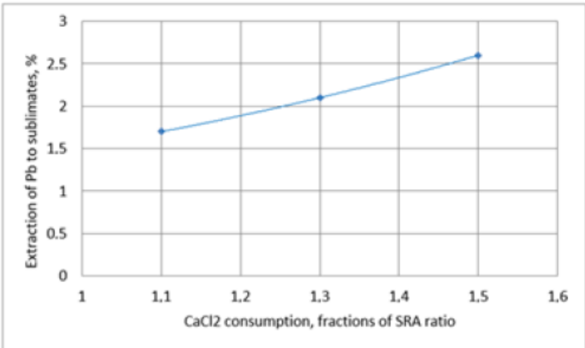
An increase in the consumption of $CaCl_2$ increases its contact with the oxides of Zn and Pb and promotes the intensification of the chlorination process, accelerating the speed and completeness of the reactions. However, it has been established that with an increase in the consumption of $CaCl_2$ (above its SRA = 1.3), the zinc content in the sublimates slows down. This is explained by the fact that when the consumption of calcium chloride is equal to SRA = 1.3, the processes of formation of zinc chloride and its sublimation are almost completed. This is evidenced by the established minimum contents of Zn, and Pb in clinker, equal to 0.92% and 0.23%, respectively, at a consumption of $CaCl_2$ = 1.5 SRA (Fig. 3 (b)). It follows that the optimal conditions for high-temperature chlorinating sintering, ensuring high extraction of zinc into sublimates, are achieved at consumption of $CaCl_2$ not exceeding its stoichiometric required amount by 1.3 times for the reduction of zinc from its oxide.

The dynamics of the extraction of lead and zinc into sublimates depending on the consumption of $CaCl_2$ is shown in Fig. 4.

Figure 4 shows that with a consumption of $CaCl_2$ 1.5 times higher than its consumption from the SRA for the reduction of zinc oxide to its chloride, a high extraction of Zn into sublimates is achieved, equal to 98.4%, with a minimum extraction of lead into sublimates - 2,6%. At the same time, a large excess of $CaCl_2$ in the process increases the extraction of volatile lead chlorides and impurities into sublimates, which will significantly reduce the quality of the resulting zinc sublimates. Based on this, and also taking into account the increase in material costs, it is advisable to take the optimal consumption of calcium chloride in the work as 1.3 times higher than the consumption from the SRA required to reduce zinc oxide to volatile zinc chloride.



a)



b)

Fig.4 - Dependence of extraction of zinc (a) and lead (b) to sublimates from $CaCl_2$ consumption: t =900 °C, τ =60 minutes

Thus, the obtained results completely confirm the previously established patterns of behaviour of zinc and lead obtained in laboratory conditions. Optimal process parameters corresponding to t =900 °C, process time - 60 minutes and $CaCl_2$ consumption 1.3 times higher than its consumption from SRA for the recovery of zinc from its oxide provide a high recovery of zinc into sublimates of more than 98%.

The yield of products at optimal parameters after sintering was, % of the weight of the charge (clinker + $CaCl_2$): clinker - 43%; sublimates – 50%, others.

High-quality zinc sublimates were obtained, wt.%: 47.39 Zn; 0.005 Pb; 51.77 Cl, others. Composition of the final clinker, wt.%: 22.53 Fe; 4.1 Si; 44 Ca; 18.26 Cl; 7.21 O; 0.26 Pb; 1.0 Zn, others. Extraction of zinc into sublimates – 98.2%.

The obtained data are in good agreement with laboratory experiments [21], which show the high reproducibility of the process under scaling conditions and the accuracy (more than 98%) of the results obtained.

The establishment of quantitative relationships between Zn, Pb, Fe and associated impurities in the final clinker has a great practical importance. To carry out the calculations, the contents of all possible impurities were established within the lower limits of their detection in the clinker obtained after sintering. Based on the results, the clinker ingredients (kg/t) were determined and their ratios were calculated, which are presented in Fig. 5.

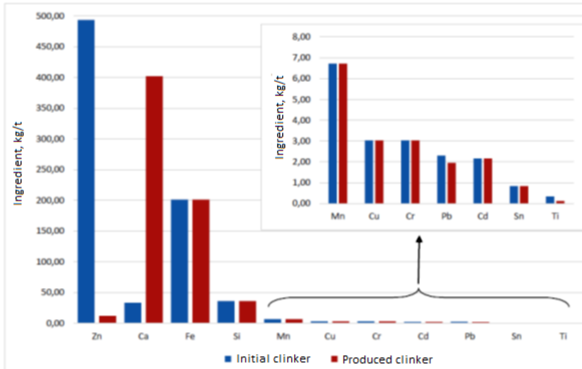


Fig.5 - Ingredients of clinker obtained after high-temperature sintering: t =900 °C, τ =60 minutes

It is easy to see that the amount of basic ingredients (Zn, Ca, Fe, Si) of clinker undergoes significant changes after sintering: the amount of Zn in clinker sharply decreases from 480 kg to ~10 kg, while the contents of Fe and Si remain almost at the same level. A significant increase in the amount of calcium in clinker from 30 kg to 400 kg is explained by the introduction of an additional amount of calcium chloride into the process as a chlorinating reagent. From the general spectrum of detected impurities, a decrease in the content of lead and titanium from their base content in the original clinker was established by 1.2 and 2.3% abs., respectively. The remaining impurities are almost completely concentrated in clinker. The obtained results show that the sublimates in the process are mainly represented by zinc chloride vapors with a minimal content of lead, titanium and tin chlorides. With further processing of the resulting zinc sublimates, their presence will not require a significant increase in costs.

Conclusions

It has been established that the minimum zinc content in clinker of 1.01% is achieved at a temperature of 900 °C. A high zinc content in sublimates of 47.42% was achieved at a consumption of $CaCl_2$ 1.3 times higher than its SRA for the reduction of zinc oxide.

The optimal parameters for high-temperature sintering have been established: t =900 °C, τ =60 minutes, $CaCl_2$ consumption is 1.3 times higher than its consumption from the SRA. High-quality zinc sublimates were obtained, wt.%: 47.39 Zn; 0.005 Pb; 51.77 Cl, others. The maximum extraction of zinc into sublimates was achieved - 98.2%.

New data were obtained on changes in the quantitative ratios of clinker ingredients under sintering conditions. It has been established that the most important ingredient (Zn) of clinker, due to the high volatility of its chloride, is concentrated in sublimates during the sintering process. Minor loss of volatile impurities is characterized by lead, titanium and tin chlorides. The remaining impurities are concentrated in clinker in the form of oxides and chlorides.

The obtained results were used in the construction of a common technology for the selective extraction of zinc and lead from ferrous metallurgy dust.

Conflict of interest. On behalf of all authors, the corresponding author confirms that there is no conflict of interest.

CRediT author statement: **N.Dosmukhamedov**: Supervision, Conceptualization, Methodology. **G.Koishina:** Data curation, Writing- Original draft preparation. **A.Argyn:** Investigation. **Yu.Icheva:** Investigation, Software, Validation: **E.Zholdasbay:** Writing- Reviewing.

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Қара металлургия шаңын кальций хлоридімен бірге күйежентектеудің кеңейтілген зертханалық тәжірибелері

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ТҮЙІНДЕМЕ
Жұмыста клинкерді $CaCl_2$-мен жоғары температурада күйежентектеу бойынша іріленген-зертханалық тәжірибелер жүргізілді. Процесті масштабтау жағдайында жоғары температуралы күйежентектеудің мақсатты өнімдеріне мырыш, қорғасын және ілеспе қоспаларды бөліп алу бойынша жаңа деректер алынды. Жоғары температуралы күйежентектеудің оңтайлы параметрлері анықталды: t=900 °C, τ=60 минут, $CaCl_2$ шығыны СҚМ-нен 1,3 есе көп. Жоғары сапалы мырыш возгондары алынды. Возгондарға мырыштың максималды бөлінуі қол жеткізілді - 98,2 %. Күйежентектеу жағдайында клинкер ингредиенттерінің сандық арақатынастары туралы жаңа мәліметтер алынды. Возгонда мырыштың хлорид түрінде болатындығы анықталды. Алынған нәтижелер қара металлургиядағы қорғасыны бар мырыш шаңын өңдеудің хлорлау технологиясының соңғы сатысының негізін құрайды және цинк пен қорғасынды тауарлық өнімге іріктеп алудың жалпы технологиясын құруда қолданылатын болады.

Түйін сөздер: шаң, қорғасын, мырыш, бөліп алу, хлорлы күйдіру.

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Укрупненно-лабораторные опыты спекания пыли черной металлургии совместно с хлоридом кальция

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АННОТАЦИЯ

В работе проведены укрупненно-лабораторные опыты по высокотемпературному спеканию клинкера с $CaCl_2$. Получены новые данные по извлечению цинка, свинца и сопутствующих примесей в целевые продукты высокотемпературного спекания в условиях масштабирования процесса. Установлены оптимальные параметры высокотемпературного спекания: t=900 °C, τ=60 минут, расход $CaCl_2$ в 1,3 раза превышающий его расход от СНК. Получены цинковые возгоны высокого качества. Достигнуто максимальное извлечение цинка в возгоны – 98,2 %. Установлены количественные соотношения ингредиентов клинкера в условиях спекания. Установлено, что цинк в виде хлорида в возгонах. Полученные результаты составляют основу заключительной стадии хлорирующей технологии переработки свинецсодержащей цинковой пыли черной металлургии и будут использованы при построении общей технологии для селективного извлечения цинка и свинца в товарные продукты.

Ключевые слова: пыль, цинк, свинец, извлечение, хлорирующее спекание.

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