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KINETICS AND INFLUENCING FACTORS IN THE CARBOTHERMIC REDUCTION PROCESS OF STEELMAKING DUST

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Abstract. *The present paper sets out the findings of a scientific investigation into the kinetics and key influencing factors of the carbothermic reduction process of steelmaking dust. Electric arc furnace dust has been found to contain significant quantities of iron and zinc oxides, which represent valuable secondary raw materials, but which also pose serious environmental risks. The present study focuses on the effects of temperature, carbon content, particle size, gas atmosphere, and reduction time on the reduction kinetics. Carbothermic reduction experiments were conducted within the temperature range of 700–1150°C, with coke serving as the reducing agent. The degree of reduction and metallization was determined through gravimetric and phase analyses. Phase composition and microstructural changes were examined using X-ray diffraction, scanning electron microscopy, and energy-dispersive spectroscopy. The experimental data were analysed using the shrinking core model to identify the rate-controlling steps of the process. The findings suggest that zinc oxide undergoes reduction more rapidly than iron oxides, a phenomenon attributable to its diminished thermodynamic stability and volatilization behaviour. The activation energies and kinetic parameters were calculated based on the Arrhenius equation. The findings provide a scientific basis for the optimisation of carbothermic reduction processes and the improvement of the efficiency of steelmaking dust recycling under industrially relevant conditions.*

Keywords: *steelmaking dust, carbothermic reduction, zinc oxide, iron oxides, kinetics, metallization degree, activation energy, gas –solid reaction, secondary raw materials, X-ray diffraction, scanning electron microscopy*

КИНЕТИКА И ФАКТОРЫ, ВЛИЯЮЩИЕ НА ПРОЦЕСС КАРБОТЕРМИЧЕСКОГО ВОССТАНОВЛЕНИЯ СТАЛЕПЛАВИЛЬНОЙ ПЫЛИ

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Аннотация. *В данной работе изложены результаты научного исследования кинетики и основных влияющих факторов процесса карботермического восстановления сталеплавильной пыли. Было обнаружено, что пыль электрических дуговых печей содержит значительное количество оксидов железа и цинка, которые представляют собой ценное вторичное сырье, но также представляют серьезную экологическую опасность. В данном исследовании основное внимание уделяется влиянию температуры, содержания углерода, размера частиц, газовой атмосферы и времени восстановления на кинетику восстановления. Эксперименты по карботермическому восстановлению проводились в диапазоне температур 700-1150 °C, при этом в качестве восстановителя выступал кокс. Степень восстановления и металлизации определялась гравиметрическим и фазовым анализом. Фазовый состав и микроструктурные изменения были изучены с помощью рентгеноструктурного анализа, сканирующей электронной микроскопии и энергодисперсионной спектроскопии. Экспериментальные данные были проанализированы с использованием модели сжимающегося ядра для определения этапов контроля скорости процесса. Результаты показывают, что оксид цинка восстанавливается быстрее, чем оксиды железа, явление, обусловленное его сниженной термодинамической стабильностью и поведением улетучивания. Энергии активации и кинетические параметры были рассчитаны на основе уравнения Аррениуса. Полученные результаты дают научную основу для оптимизации процессов карботермического восстановления и повышения эффективности переработки сталеплавильной пыли в промышленно-актуальных условиях.*

Ключевые слова: *сталеплавильная пыль, карботермическое восстановление, оксид цинка, оксиды железа, кинетика, степень металлизации, энергия активации, газо-твердая реакция, вторичное сырье, Сканирующая электронная микроскопия, энергодисперсионная спектроскопия*

PO‘LAT ERITISH PECHI CHANGLARINI KARBOTERMIK TIKLASHDA JARAYONNING KINETIKASI VA TA’SIR ETUVCHI OMILLAR

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Annotatsiya. *Ushbu ishda po‘lat eritish changini karbotermik tiklash jarayonining kinetikasi va asosiy ta’sir etuvchi omillarini ilmiy o‘ganish natijalari bayon etilgan. Elektr yoy pechi changida ko’p miqdorda temir va rux oksidlari mavjudligi aniqlangan, ular qimmatli ikkilamchi xomashyo hisoblanadi, ammo atrof-muhit uchun jiddiy xavf tug‘diradi. Ushbu tadqiqot harorat, uglerod miqdori, zarrachalar o‘lchami, gaz atmosferasi va tiklanish vaqtining tiklanish kinetikasiga ta’siriga qaratilgan. Karbotermik qaytarish tajribalari 700-1150 °C harorat oralig‘ida olib borildi, qaytaruvchi sifatida koks ishlatildi. Qaytarilish va metallizatsiya darajasi gravimetrik va fazaviy tahlillar orqali aniqlandi. Fazaviy tarkib va mikrostrukturaviy o‘zgarishlar rentgenografik, skanerlovchi elektron mikroskopiya va energetik-dispersion spektroskopiya usullari yordamida o’rganildi. Jarayonning tezlikni nazorat qilish bosqichlarini aniqlash uchun eksperimental ma’lumotlar qisqaruvchi yadro modeli yordamida tahlil qilindi. Natijalar shuni ko‘rsatadiki, rux oksidi temir oksidlariga qaraganda tezroq tiklanadi, bu uning termodinamik barqarorligi va uchish xususiyatining pasayishi bilan bog‘liq. Arrenius tenglamasi asosida aktivlanish energiyalari va kinetik parametrlari hisoblandi. Olingan natijalar sanoat uchun dolzarb sharoitlarda karbotermik tiklash jarayonlarini optimallashtirish va po’lat eritish changini qayta ishlash samaradorligini oshirish uchun ilmiy asos bo‘lib xizmat qiladi.*

Kalit so‘zlar: *po’lat eritish changi, karbotermik tiklanish, rux oksidi, temir oksidlari, kinetika, metallizatsiya darajasi, faollanish energiyasi, gaz-qattiq reaksiya, ikkilamchi xomashyo, skanerlovchi elektron mikroskopiya, energiya dispersion spektroskopiya*

Introduction

The rapid development of the modern metallurgical industry is leading to a sharp increase in steel production volumes. In electric arc furnaces (EAF), converters, and other high-temperature units, large amounts of dust-like waste are generated during steel smelting processes. This steelmaking dust (Electric Arc Furnace Dust - EAFD) contains, along with iron and zinc oxides, non-ferrous and heavy metals such as lead, chromium, and manganese, which are, on the one hand, valuable secondary raw materials, and on the other hand, a source of environmental hazards [1,2].

Scientific research shows that the content of iron oxides (FeO, Fe_2O_3) in steel smelting dust can reach 30-50%, and the proportion of zinc oxide (ZnO) in some cases can reach 15-35% [3,4]. Therefore, the recovery of metal resources and the reduction of waste through the processing of this dust is one of the priority tasks of the metallurgical industry. Traditional hydrometallurgical and pyrometallurgical technologies are characterized by high reagent consumption, multi-stage processes, and environmental limitations [5,6].

In recent years, the carbon-thermal reduction technology for processing steelmaking dust has gained special attention due to its high efficiency, technological simplicity, and industrial applicability [7-9]. The main advantage of this method is that at high temperatures, zinc oxide is first reduced and released in vapor form, while iron oxides transition to a metallic state. This allows for selective dezincification and metallization of iron [10,11].

Despite the thermodynamic favorability of the carbon-thermal reduction process, its effectiveness under actual industrial conditions is primarily determined by kinetic factors. Several researchers have demonstrated that the reduction of ZnO and FeO in the presence of carbon or CO is a multi-stage gas-solid phase process [12-14]. The process rate is limited by CO formation through the Boudouard reaction, gas diffusion, chemical reaction, and the removal of products from the system [15].

It has been widely reported in the literature that the kinetics of carbon-thermal reduction of ZnO proceed faster than that of FeO. The main reason for this is the lower thermodynamic stability of ZnO and the volatility of zinc [16,17]. Studies conducted by Chen et al. have determined that the reduction activation energy of ZnO is in the range of 60-80 kJ/mol, while for FeO this value is 90-110 kJ/mol [1,18]. This provides scientific evidence for the selective reduction of zinc oxide.

The Shrinking Core Model is widely used in the mathematical description of the reduction process. This model takes into account the preservation of an unreduced core within the particle and the movement of the reaction front towards the center over time [19]. The correlation of experimental data with this model allows for the identification of rate-limiting stages of the process and the establishment of optimal technological parameters.

Furthermore, several studies have shown that factors such as temperature, carbon content, particle size, gas environment composition, and reduction time significantly affect the reduction kinetics [8,9,20]. An increase in temperature intensifies the Boudouard reaction, raises CO concentration, and activates diffusion processes, leading to a sharp increase in the degree of metallization.

However, the literature review indicates that a comprehensive kinetic analysis of the carbothermic reduction process of steelmaking dust, specifically the simultaneous reduction of ZnO and FeO, their interaction, and an in-depth scientific substantiation of the rate-limiting stages, has not been adequately addressed. In particular, the determination of kinetic parameters for process optimization under near -industrial conditions remains a pressing scientific challenge.

The purpose of this article is to conduct an in-depth study of the kinetic regularities of the carbon-thermal reduction process for steelmaking dust, identify the reduction mechanisms of FeO and ZnO, and scientifically substantiate the role of the main technological factors influencing the process.

Materials and methods

Steel smelting dust generated during the steel smelting process in an electric arc furnace was selected as the object of research. Dust samples were collected from gas purification systems under industrial conditions, dried in laboratory conditions, and purified from mechanical impurities. The average particle size of the samples ranged from 10 to 100 μm, and their finely dispersed structure exhibited a high surface area.

The chemical analysis was conducted using the X-ray fluorescence (XRF) technique. The results of the study are summarised in Table 1.

Table 1

Chemical composition of EAF dust (wt.%)

Component	Fe_{total}	Fe_2O_3	FeO	ZnO	SiO_2	CaO	Al_2O_3	MnO	PbO	Na_2O
Content	31.2	41.6	4.6	16.2	3.4	3.4	0.9	3.7	1.36	10.4

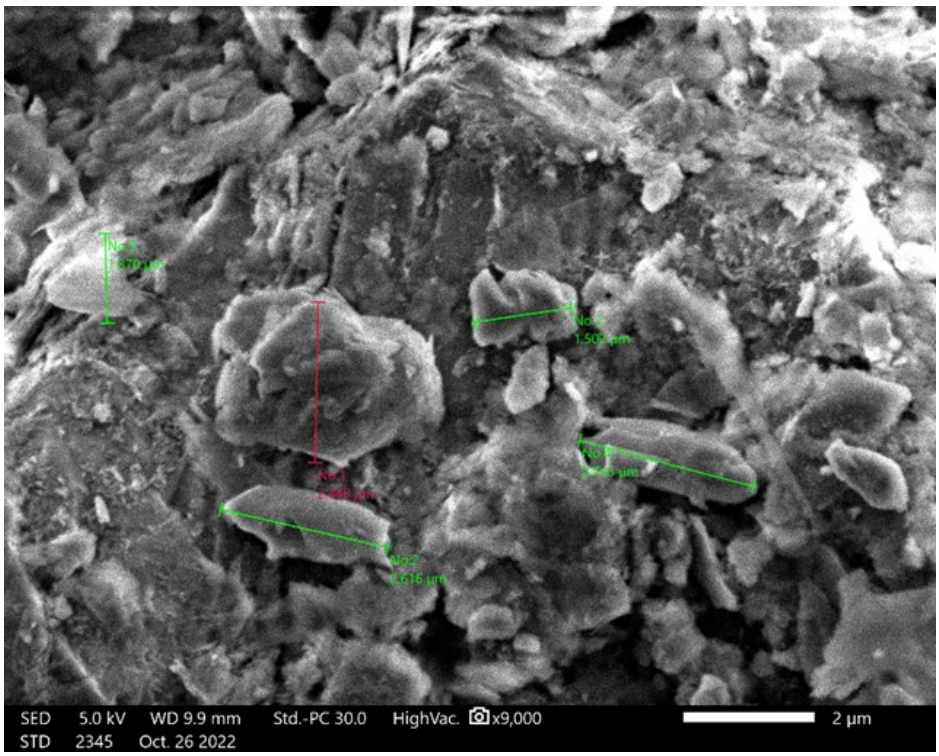

Fig. 1. Initial SEM microstructure of steel smelting dust

The irregular shape and high porosity of dust particles create favorable conditions for the reduction process.

Scanning electron microscopy (SEM) images of dust particles obtained from the steelmaking furnace gas cleaning system reveal their irregular shape, high porosity, and wide particle size distribution. These structural features increase the specific surface area of the particles and create favorable conditions for gas-solid phase reactions during the carbon-thermal reduction process. The porous structure facilitates the diffusion of CO gas into the inner part of the particle, leading to an acceleration of the reduction kinetics.

The chemical composition of the dust was determined using X-ray fluorescence (XRF) and energy-dispersive spectral analysis (EDS) methods. According to the research results, the main components in the dust composition are FeO, Fe_2O_3, and ZnO, with zinc oxide content around 15-30% and the total share of iron oxides constituting 40-55%. Additionally, small amounts of CaO, SiO_2, and MgO were detected.

High-purity carbon coke powder was used as the reducing agent. The ash content of the carbon did not exceed 8%, and the particle size was selected in the range of 50-80 µm. The carbon content was varied between 5-15% relative to the dust mass.

Carbon-thermal reduction experiments were conducted under laboratory conditions in a tubular electric furnace. The dust and carbon mixture was blended in predetermined ratios and placed in cylindrical ceramic crucibles. The experiments were carried out in an inert or controlled gas environment.

The temperature was varied in the range from 700 to 1150 °C, with 50 °C increments. In each experiment, the sample was held at the given temperature for 30-60 minutes. The heating rate was set at 10 ° C/min. At the end of the experiment, the samples were rapidly cooled and prepared for subsequent analysis.

To determine the time dependence of the reduction process, experiments were repeated at various time intervals. This method allowed for comparative analysis of the reduction kinetics of ZnO and FeO.

The phase composition of the reduced products was determined using X-ray diffraction (XRD) analysis, and the degree of transition of oxides to metallic state was assessed. The microstructure of the initial and reduced samples was studied using a scanning electron microscope (SEM), and the distribution of elements by phases was determined using EDS.

The degree of metallization was calculated using the following expression:

$$M = \frac{Fe_{met}}{Fe_{total}} * 100\% \quad (1)$$

where Fe_{met} is the amount of iron in metallic state, Fe_{total} is the total amount of iron in the sample.

Kinetic analysis of the reduction process was carried out by determining the dependence of the degree of reduction (α) on time and temperature. The degree of reduction was calculated using the following formula:

$$\alpha = \frac{m_0 - m_t}{m_0 - m_{final}} \quad (2)$$

where m_0 is the initial mass, m_t is the mass at a given time, m_{final} is the mass after complete reduction.

The obtained experimental data were analyzed based on the Shrinking Core Model. The following kinetic equations were used to determine the rate-limiting stage of the process:

for the case limited by chemical reaction:

$$1-(1-a)1/3 = kt \quad (3)$$

for the case limited by diffusion:

$$1-\frac{32(1-a)2}{3}+12(1-a) = kt \quad (4)$$

The reaction rate constants were determined for different temperatures, and the activation energy was calculated using the Arrhenius equation:

$$k = A\exp(\frac{E_a}{RT}) \quad (5)$$

Graphs were constructed in ln k vs. 1/T coordinates, and kinetic parameters were evaluated based on linear relationships.

The influence of factors such as temperature, carbon content, particle size, gas environment, and reduction time on the reduction process was studied separately. When each factor was changed, the remaining parameters were kept constant. This approach allowed for the identification of individual impact mechanisms of the process.

Each experiment was repeated at least three times. The average values and deviations of the obtained results were calculated, ensuring that the experimental error did not exceed 5%.

Results

Laboratory experiments have shown that the process of carbothermic reduction of steelmaking dust has a complex, multi-stage kinetic mechanism. It was found that the behavior of ZnO and FeO oxides during the reduction process differs significantly, which is explained by differences in their thermodynamic stability and kinetic parameters.

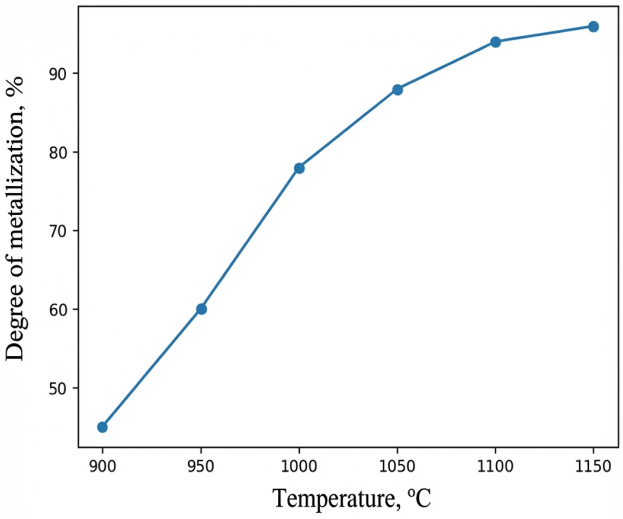

Fig. 2. Influence of temperature on the degree of metallization

Analysis of the dependence of the reduction degree (α) on time showed that in the initial stage of the process, the reduction of ZnO occurs very rapidly, while the reduction of FeO occurs relatively slowly but steadily. This circumstance confirms the existence of a selective dezincification mechanism.

A sharp increase in the degree of metallization in the range of 1000-1150 °C confirms the presence of an optimal temperature range for the reduction process.

The figure shows the effect of temperature on the degree of metallization. As the temperature increases from 900°C to 1150°C, the degree of metallization rises from 45% to 96%. A sharp increase in the degree of metallization, particularly in the range of 1000-1150°C, indicates the existence of an optimal temperature range for the reduction process.

It was determined that an increase in temperature has a decisive impact on the speed and completeness of the reduction process. In the range of 800-900°C, the reduction of ZnO begins, and zinc is released in vapor form. At temperatures above 1000°C, active reduction of iron oxides was observed.

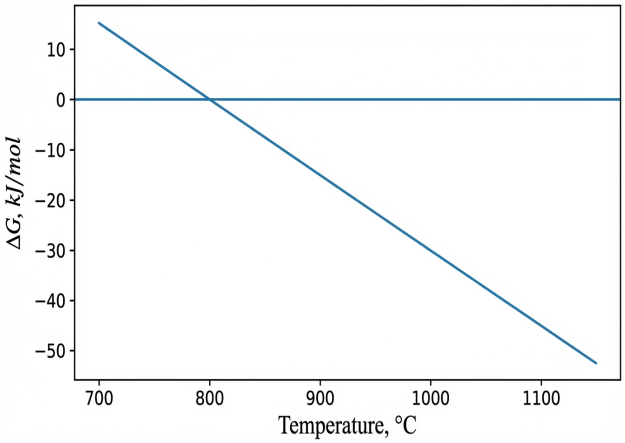

Fig. 3. Temperature dependence of Gibbs free energy for the reduction reaction of ZnO in the presence of carbon

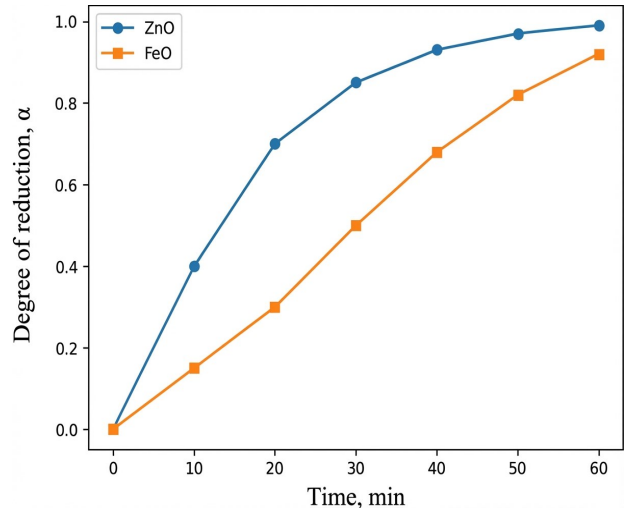

Fig. 4. Dependence of the degree of reduction (α) on time. The rapid reduction of ZnO and the slower reduction of FeO confirm the mechanism of selective dezincification

At temperatures above 800°C, ΔG < 0 indicates thermodynamically favorable reduction of zinc oxide.

The temperature dependence of Gibbs free energy for the reaction ZnO + C → Zn + CO is shown in the figure. As evident from the graph, at temperatures above 800°C, Gibbs free energy transitions to negative values (ΔG < 0), which indicates that the reduction of zinc oxide with carbon is thermodynamically favorable.

The figure shows the dependence of the reduction degree of ZnO and FeO on time during the carbothermic reduction process. The reduction of ZnO occurs rapidly in the first 20-30 minutes, reaching α ≈ 0.85-0.90. The reduction of FeO is relatively slower, reaching a value of α ≈ 0.90-0.95 in 60 minutes.

In the temperature range of 1000-1150 °C, the degree of metallization increased sharply, reaching 90-96%. This temperature range indicates the thermodynamically and kinetically most favorable region for the carbothermic reduction process.

Discussion

These results are consistent with the research conducted by Chen and Pickles, who also observed the reduction of ZnO at lower temperatures compared to FeO.

As temperature increases, the equilibrium of the Boudouard reaction shifts to the right, increasing the concentration of CO. This leads to the acceleration of chemical reactions at the gas-solid interface and the enhancement of diffusion processes.

It was found that when the carbon content was varied between 5-15% of the dust mass, the reduction efficiency differed significantly. At a carbon content of 10-15%, the reduction of ZnO and FeO occurred at the highest level.

With insufficient carbon, CO formation is limited, and the degree of reduction decreases. Conversely, excess carbon led to increased energy consumption and higher residual carbon in the product, without significantly increasing the degree of metallization.

This indicates that the reduction process is mainly associated with the Boudouard reaction. The optimal amount of carbon ensures a balance between CO formation and oxide reduction.

The reduction degree-time relationships were analyzed based on the shrinking core model. Experimental points for the ZnO reduction process fit well with the model limited by diffusion and evaporation. It was determined that the reduction of FeO is mainly limited by the chemical reaction at the gas-solid phase interface.

An increase in the coke content significantly improved zinc removal due to the intensification of both direct solid-phase reactions and indirect gas-phase reduction mechanisms. The experimental results are summarized in Table2.

Table 2

Effect of Coke Content on Residual Zinc in Clinker

Coke content, wt.%	Residual Zn, wt.%
52	13,4
65	10,56
80	7,98
90	7,98

The activation energies calculated based on Arrhenius plots are as follows:

For ZnO reduction: E_a=60-80 kJ/mol;

For FeO reduction: E_a=90-110 kJ/mol.

The lower activation energy of ZnO and the evaporation property of zinc ensure its faster reduction. The reduction of FeO occurs more slowly due to the high activation energy. This scientifically substantiates the mechanism of selective zinc removal.

In a CO-rich gas environment, the reduction rate increased significantly. The main part of ZnO was reduced in 30-40 minutes, while 40-60 minutes were required for complete reduction of FeO.

It was found that open or low-pressure systems are more effective, as the increase in pressure leads to partial condensation of zinc vapors.

The obtained results demonstrated that the carbon-thermal reduction process of steelmaking dust is effective for selective dezincification and obtaining iron products with a high degree of metallization under industrial conditions. Under optimal conditions, zinc can be released in vapor form and subsequently collected through condensation, while iron is reused as a secondary raw material in metallurgical processes.

Conclusion

In this study, the thermodynamic and kinetic principles of the carbon-thermal recovery process for dust generated during steel smelting were comprehensively examined. Experimental and theoretical analyses revealed that the reduction of iron and zinc oxides in steel smelting dust occurs through a multi-stage gas-solid phase mechanism.

The research results showed that the carbon-thermal reduction of ZnO begins at lower temperatures and proceeds faster compared to FeO. This phenomenon is attributed to the lower thermodynamic stability of zinc oxide, zinc's volatility, and its relatively low activation energy. The reduction of iron oxides primarily intensifies at high temperatures and culminates in the formation of metallic iron.

Based on kinetic analysis, it was determined that the ZnO reduction process is mainly limited by diffusion and zinc evaporation, while the FeO reduction process is limited by the chemical reaction at the gas-solid interface. The activation energies calculated using the Arrhenius equation confirmed these conclusions and demonstrated that the process follows Arrhenius's law.

It was found that an increase in temperature significantly enhances the rate and completeness of the reduction process by accelerating the Boudouard reaction, resulting in increased CO concentration. Optimal reduction conditions were achieved in the temperature range of 1000-1150 °C, with the degree of metallization reaching 90-96%. The optimal range of carbon content was determined to be 10-15% of the dust mass, and it was shown that higher amounts decrease energy efficiency.

Particle size and structural properties were found to significantly influence the reduction kinetics, with higher reduction rates observed in fine-dispersed dust. However, the need for granulation to ensure process stability under industrial conditions was substantiated. The composition of the gas environment and reduction time were also found to directly affect process efficiency, with a notable increase in reduction rate observed in a CO-rich environment.

The results of the conducted research demonstrated that the carbon-thermal reduction process of steelmaking dust is scientifically and technologically feasible for selective dezincification and obtaining iron products with a high degree of metallization. The obtained kinetic and technological regularities serve as a solid scientific foundation for developing resource-saving and environmentally safe technologies for processing metallurgical waste.

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