



Chair of Mining Engineering and Mineral Economic

Master's Thesis



Hydrometallurgical Recovery of Zinc  
from Blast Furnace Sludge

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Juli 2023



**AFFIDAVIT**

I declare on oath that I wrote this thesis independently, did not use other than the specified sources and aids, and did not otherwise use any unauthorized aids.

I declare that I have read, understood, and complied with the guidelines of the senate of the Montanuniversität Leoben for "Good Scientific Practice".

Furthermore, I declare that the electronic and printed version of the submitted thesis are identical, both, formally and with regard to content.

Date 03.07.2023

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Signature Author  
Galina Iureva

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## Acknowledgement

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## **Abstract**

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In light of the increasing need for recycling secondary sources of metals, one of the most pressing challenges is the extraction of zinc from blast furnace sludge (BFS). The sludge cannot be reintroduced into the blast furnace as it negatively affects the smelting process. Various methods of sludge processing were explored to reduce environmental impact regenerating components and recover valuable metals from the sludge. Thorough analyses of the sludge's mineralogical and chemical composition were conducted to determine the optimal method and leaching reagents for zinc recovery. The research demonstrated the effectiveness of hydrochloric acid leaching in achieving a high zinc recovery rate. The developed closed-loop circuit not only focuses on zinc recovery but also facilitates the recovery of the reagents used in the process. Implementing this circuit contributes to the development of a sustainable recycling system for blast furnace sludge, promoting efficient zinc recovery and reducing waste generation. The findings have implications for the iron and steel industry, fostering a more environmentally friendly and economically viable approach to waste management, resource recovery, and reagent utilization.

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## Zusammenfassung

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Im Hinblick auf den zunehmenden Bedarf an Recycling von sekundären Metallquellen stellt die Extraktion von Zink aus dem Hochofenschlamm (BFS) eine der dringendsten Herausforderungen dar. Der Schlamm kann nicht in den Hochofen zurückgeführt werden, da er den Schmelzprozess negativ beeinflusst. Es wurden verschiedene Methoden zur Schlammverarbeitung erforscht, um die Umweltauswirkungen zu verringern, Komponenten zu regenerieren und wertvolle Metalle aus dem Schlamm zurückzugewinnen. Gründliche Analysen der mineralogischen und chemischen Zusammensetzung des Schlammes wurden durchgeführt, um die optimale Methode und Auslaugungsmittel für die Zinkrückgewinnung zu bestimmen. Die Forschung hat die Wirksamkeit der Auslaugung mit Salzsäure bei der Erzielung einer hohen Zinkrückgewinnungsrate gezeigt. Der entwickelte geschlossene Kreislauf konzentriert sich nicht nur auf die Zinkrückgewinnung, sondern ermöglicht auch die Rückgewinnung der im Prozess verwendeten Auslaugungsmittel. Die Umsetzung dieses Kreislaufs trägt zur Entwicklung eines nachhaltigen Recycling-Systems für Hochofenschlamm bei, das eine effiziente Zinkrückgewinnung fördert und die Abfallerzeugung reduziert. Die Ergebnisse haben Auswirkungen auf die Eisen- und Stahlindustrie und fördern einen umweltfreundlicheren und wirtschaftlich tragfähigeren Ansatz für die Abfallwirtschaft, die Ressourcenrückgewinnung und die Verwendung von Auslaugungsmitteln.

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# 1 Introduction

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In recent years, there has been a growing global concern about environmental sustainability, leading to increased scrutiny of industries due to their significant waste generation and environmental impact. Among these industries, the iron and steel sector face a notable environmental challenge with blast furnace sludge (BFS).

BFS, a by-product of iron and steel production, is composed of fine particles, metal oxides, and carbonaceous materials. Apart from its high iron content, BFS is also characterized by elevated levels of zinc. The presence of zinc in sludge presents challenges for its direct return to the base cycle in blast furnace production, as it contributes to lining deterioration, scaling, and increased coke consumption.

Therefore, BFS is usually stored in sludge storage facilities. Its accumulation presents various challenges, including land and water pollution, as well as the depletion of natural resources. Conventional disposal methods like landfilling not only fail to address these issues but also worsen the environmental burden. Consequently, there is an urgent need to explore sustainable alternatives for sludge processing.

Furthermore, unlike traditional ores that necessitate mining and extensive processing, BFS presents a distinct cost-efficient advantage. The valuable metals present in the sludge offer an opportunity for resource recovery without incurring additional extraction or processing expenses.

The objective of this master's thesis is to investigate and propose an environmentally sound technology for processing BFS. The study will analyse the environmental impact of BFS disposal practices, evaluate existing recycling methods, and explore innovative techniques to enhance the efficiency and sustainability of BFS processing. The research will encompass a comprehensive review of existing literature, case studies of BFS processing facilities, and laboratory experiments to assess the viability and effectiveness of proposed recycling techniques.



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## 2 Theoretical background and literature study

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### 2.1 Overview of the zinc industry in Russia

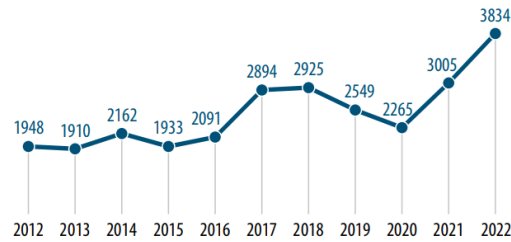
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Zinc has a multitude of applications. It provides corrosion protection for diverse steel components and structural elements and facilitates the creation of zinc alloys like brass, bronze, and die casting alloys. Zinc-based semi-finished goods find use in construction, automotive, and appliance sectors. On the global scale for non-ferrous metal demand, zinc ranks third, following aluminium and copper.

Russia holds a leading position in global zinc reserves, ranking second behind China. Its primary source of zinc is copper-pyrite deposits, complemented by metalliferous-polymetallic facilities, although the role of stratiform lead-zinc deposits has increased in recent years. Despite the relatively low metal content of its deposits, Russia maintains a quality comparable to international standards. (VIMS *et al.*, 2022) Additionally, the country ranks among the top ten global producers of zinc concentrate and metal. (Reichl and Schatz, 2023)

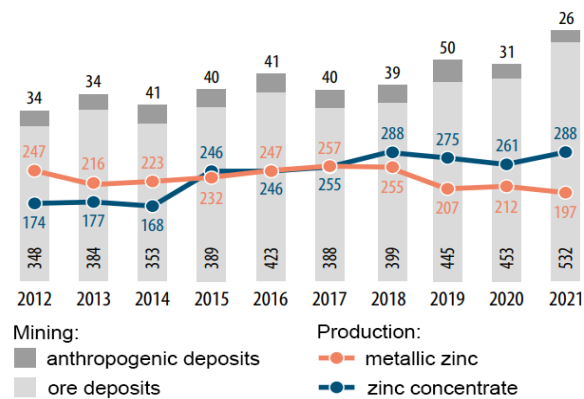
Zinc prices between 2011 and 2016 were relatively stable, influenced by overproduction and reduced consumption in China. A significant decrease in reserves between 2016–2018 led to a temporary price rise, offset by resumed mining operations globally. The end of 2019 saw a 13% drop in prices on the London Metal Exchange (LME), exacerbated by a global economic slowdown and the US–China trade stand-off. The COVID-19 pandemic in 2020 further decreased the average LME price by 11.1%. However, 2021 saw a recovery, with a steady price increase of 11.5%, driven by global economic recovery, a weaker dollar, and potential raw material shortages. Despite a downward trend in May 2022, the LME zinc price for the first half of the year remained 27.6% higher than 2020's average. (VIMS *et al.*, 2022) Figure 1 shows the trend in annual average price.

Zinc metal production in Russia rose by 2.5% to 211.8 thousand tons in 2020, but experienced a 7.5% reduction to 197 thousand tons in 2021 (see Figure 2). This deviation from the preceding upward trend likely stems from a decline in overall economic activity due to the impact of the coronavirus pandemic.



**Figure 1: Dynamics of the annual average settlement price for Special High Grade (SHG) zinc from 2012 to 2021, USD/Tonne<sup>1</sup> (VIMS et al., 2022)**

Apparent domestic consumption of zinc metal has grown by 11.3% to 246,900 tonnes over the past decade, fueled by the active construction of residential and commercial real estate, as well as road and energy infrastructure development and modernization. Galvanized steel products (62%) and hot-dip galvanized steel structures (21%) predominantly constitute Russia's domestic zinc consumption, with the remaining coming from the chemical industry, non-ferrous metal processing sector, and others. The commencement of new galvanized steel production projects and the expansion of existing facilities lay the groundwork for further domestic zinc consumption growth. A new smelting facility in Verkhny Ufaley is under construction to compensate for the lost capacity following Electrozinc's 2019 closure. Polymet Engineering continues to build a zinc electrolysis plant with an annual metal capacity of 120 thousand tons, with commissioning scheduled for October 2023. (VIMS et al., 2022)



**Figure 2: Dynamics of zinc mining and production from 2012 to 2021 (in 1000 tonnes) (cp. VIMS et al., 2022)**

In 2021 Russian mineral processing plants generated 615.5 thousand tonnes of zinc concentrates (VIMS et al., 2022). The Ural Mining and Metallurgical Company and Russian Copper Company holdings produce concentrates, which are processed by Chelyabinsk Zinc Plant (CZP), Russia's sole zinc metal producer and part of UMMC.

<sup>1</sup> For 2012–2022 – annual average price, for 2022 – average price for the first half of the year

Zinc concentrates from other Russian firms are exported to Southeast Asia and Kazakhstan.

At CZP, 48% of the processed concentrates originate from Russian producers, and secondary zinc-containing raw materials account for 12%. The company faces considerable risks due to potential adverse shifts in raw material markets, as it directly relies on the supply and pricing of zinc concentrates (Chelyabinsk Zinc Plant, 2023). However, importing concentrates from Kazakhstan proves more economically viable than transporting raw materials from Russia's eastern regions (VIMS *et al.*, 2022).

Major mining and metallurgical complexes in the old mining regions are experiencing a depletion of primary mineral reserves, even as unused technogenic resources continue to expand. The potential for the increasing raw-metal base is limited due to the major reserves residing in low-zinc hard-enriched polymetallic ores or in remote areas with challenging climatic and geological conditions, such as Buryatia and Yakutia. To address this, interest in technologies for processing secondary raw materials is increasing. Incorporating unused technogenic resources into economic activity not only helps address supply issues but also mitigates environmental problems, particularly in regions with significant environmental pollution from waste storage facilities.

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## **2.2 Characteristics of the main types of zinc-containing wastes from steel industry**

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Fine-dispersed waste in the black metallurgy industry comprises dust and sludge generated from gas cleaning facilities and aspiration devices. These waste materials are residues resulting from raw materials and their processing during metallurgical production processes. The primary categories of dust wastes are detailed in Table 1.

Metallurgical dust and sludge fall under the classification of iron-containing waste, categorized based on their iron content as follows:

- Rich – with an iron content of 55–67% (sludge and dust from converter gas cleaning systems).
- Relatively rich – with an iron content of 40–55% (sludges and dust from agglomeration plants, gas cleaning systems, and subbunker areas of blast furnaces).
- Poor – with an iron content below 40% (sludge and dust from electric arc furnace gas cleaning systems).

Components	Agglomeration plant	Blast furnace	Converter	Electric arc furnace
Fe <sub>total</sub>	22–58	40–55	41–67	30–55
Fe <sub>2</sub> O <sub>3</sub>	27–70	20–60	39–78	46–63
FeO	3–22	5–9	5–67	5–46
C	1.78–12.06	6–26	0.7–4.32	0.5–3.0
Zn	0.01–0.09	0.5–15.4	0.18–4.16	> 30.0
CaO	2–21	4–10	3–20	1.5–17.0
SiO <sub>2</sub>	4–10	6–11	1,4–2,9	2–12
Al <sub>2</sub> O <sub>3</sub>	1.5–3	2–4	0.11–0.36	0.3–10.0
MgO	1–6	1–3	0.15–1.48	5.0–27.0
K <sub>2</sub> O	0.09–0.22	0.12–0.53	0.1–0.26	0.3–1.0
Na <sub>2</sub> O	0.05–0.16	0.1–0.3	0.1–0.61	1.0–2.0
Mn	0.08–0.41	0.1–0.5	0.74–1.68	1.5–5.5
P	0.02–0.27	0.03–0.23	0.04–0.18	0.02–0.25
S	0.14–0.78	0.2–0.9	0.04–0.32	0.02–0.5
Ni	0.004–0.079	0.01–0.03	0.01–0.04	0.02–8.0
Cu	0.01–0.04	0.01–0.02	0.01–0.07	0.1–0.5
Pb	0.01–0.10	0.1–0.6	0.01–0.61	> 5.0

**Table 1: Chemical composition of ferrous metallurgy dusts and sludges [wt. %]**

The chemical composition of dust and sludge varies depending on the raw material composition and the specific technological processes employed. Consequently, it can exhibit significant variations across different plants and periods of time. (Hleis *et al.*, 2013). Table 2 presents the primary dust sources from each production unit along with the corresponding dust emission factors.

Plant	Operation	Dust emission factor	Unit
Sintering	Mixing of raw material	0.5–37.7	[g/t sinter]
	Strand operation	40.7–559.4	
	Hot screening	14.5–40	
	Cooling and cold screening	14–212	
Blast furnace	Burden preparation	2–54	[g/t hot metal]
	Charging of raw materials	2.7–81.4	
	Casting house	0.42–41.95	
	Hot stove	0.4–18	
Steelmaking furnace	Pre-treatment of hot metal	1	[g/t liquid steel]
	Charging/tapping	2–60	
	O <sub>2</sub> blowing	0.3–55	
	Roof emissions	8–120	
	Secondary metallurgy	0.1–50	

**Table 2: Production units, emission sources and dust factors from the integrated iron and steelmaking process (Hleis *et al.*, 2013)**

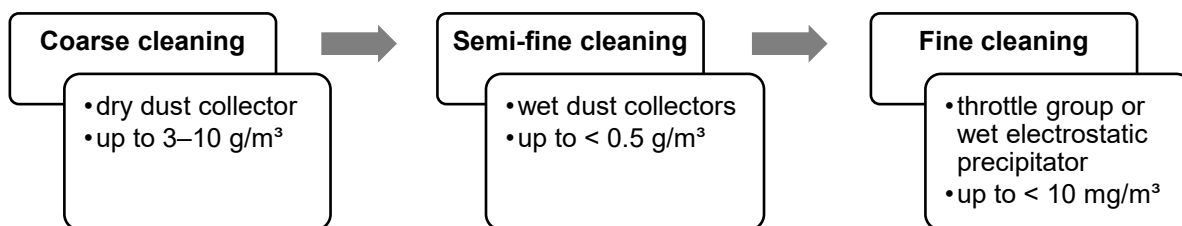
As reported by the Federal Service for Supervision of the Use of Natural Resources (2022), the accumulated BFS reached 3.4 million tonnes by 2021, with an additional

production of 374,500 tonnes during the year. Out of this total, 259,000 tonnes were recycled, while 105,700 tonnes were supplied to consumers.

Blast furnace and steelmaking dust and sludge are valuable sources of raw materials for both black and non-ferrous metals. However, the global recycling rate for zinc-containing metallurgical dust is currently only 45–50% (Bryantseva *et al.*, 2013). To make the dust and sludge suitable for further processing in non-ferrous metallurgical plants, pre-enrichment with zinc and other non-ferrous metals is necessary, reaching economically viable concentrations for established utilization methods.

### 2.3 Blast furnace sludge formation

The blast furnace generates up to 2500 m<sup>3</sup>/ton of blast furnace gas, depending on the type of pig iron produced (ITS 26-2022, 2022). The raw blast furnace gas undergoes a multi-stage purification process to remove dust before being returned to the plant network (see Figure 3).



**Figure 3: Multi-stage blast furnace gas purification process**

Initially, the dry dust collector separates a significant portion of coarse dust particles larger than 50 µm (coarse cleaning). Subsequently, the wet gas cleaning system (scrubbers or Venturi scrubbers) separates finer dust particles from the blast furnace gas (semi-fine cleaning). In the third stage (throttle group), highly dispersed fractions are separated (fine cleaning), and these fractions, in the form of sludge, are directed to radial settling tanks. The purified gas is directed to the clean gas collector.

The dust captured in the wet gas cleaning system is sent as sludge to settling basins for thickening and subsequent pumping from the bottom of the thickener. The clarified water is recycled in the water supply system. The sludges from the wet gas cleaning system of the blast furnace process have an elevated concentration of zinc and alkalis, limiting their potential for recycling. Typically, this sludge is removed from the circulation and either deposited in settling ponds or stored in sludge repositories.

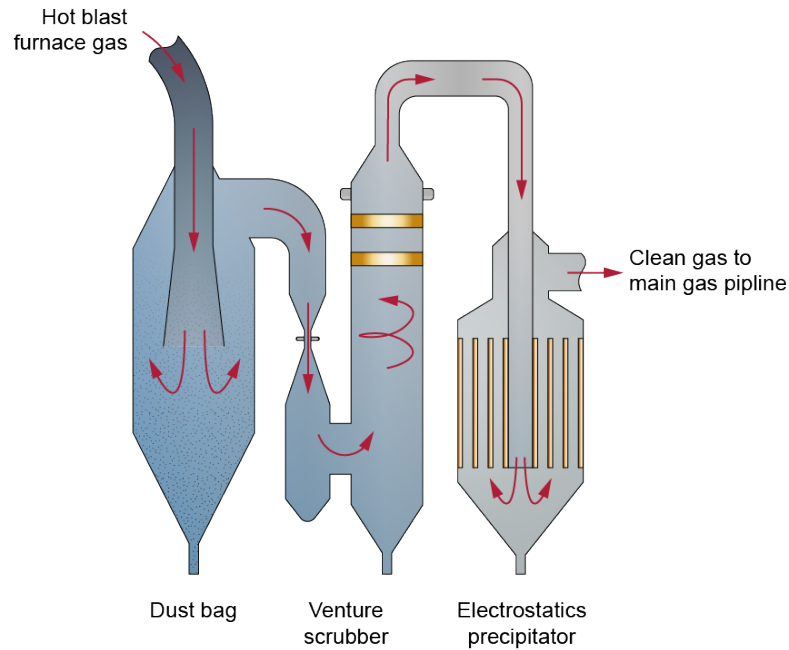


Figure 4: The flow of blast furnace gas purification (cp. Imamovic and Goletic, 2013)

## 2.4 Methods for recycling zinc-containing ferrous dust

Various technologies exist for extracting zinc from blast furnace and steelmaking dust and sludge, which can be classified into the following groups: mechanical (hydrocyclones, scrubbing), hydrometallurgical (leaching), pyrometallurgical, plasma, and liquid phase reduction processes.

Over the past 30 years, significant technological advancements based on hydrometallurgical and pyrometallurgical principles have been the primary focus of research in this field. Hydrometallurgical methods involve leaching zinc, lead, and other non-ferrous metals using acid or alkali solutions, followed by precipitation through chemical or electrochemical means. Some plants in Italy and the United States have implemented hydrometallurgical processes for the treatment of zinc-containing dust. However, the inefficiency of hydrometallurgical methods lies in their inability to extract zinc from the insoluble zinc ferrite, which is the predominant zinc compound found in metallurgical dust.

### 2.4.1 Mechanical methods

The common practice involves using hydrocyclones to process dust and sludge before incorporating them into agglomerates. This method effectively concentrates zinc in the top product, reducing zinc input into the blast furnace.

British Steel developed a stocker dust processing technique utilizing hydrocyclones. After conducting experiments at the Scunthorpe facility in December 1993, an industrial plant was established at the Llanwern facility in April 1994, followed by another plant in Teesside (Butterworth, Linsley and Aumonier, 1996).

Rasa Corp., a Japanese company, developed the DeZn method, which includes classifying BFS in a vacuum hydrocyclone. For treating dust and sludge with high zinc content, Rasa developed the Rasa-NGP process. This process utilizes a hydrocyclone and a specialized unit for scraping the concentrated surface layer containing zinc and other impurities. The Voltex scalping apparatus is used for scraping the surface layer, primarily consisting of zinc and lead compounds. Subsequently, the sludge is efficiently classified in a specially designed hydrocyclone called hydro-negaclone. The hydrocyclone operates under vacuum created by a pump. During classification, fine particles containing 80% zinc are discharged with the overflow, while the sediment, representing 75–80% of the solid material, contains a low zinc content product. The capital and operational costs of this sludge preparation method are 10–15 times lower compared to the production of metallicized pellets from waste. Japan has constructed six plants using this approach, with each plant capable of processing 120,000 tons of raw material annually (e.g., Nippon Steel, Nippon Kokan, etc.) (Korneev, Urbanovich and Rozenberg, 2021).

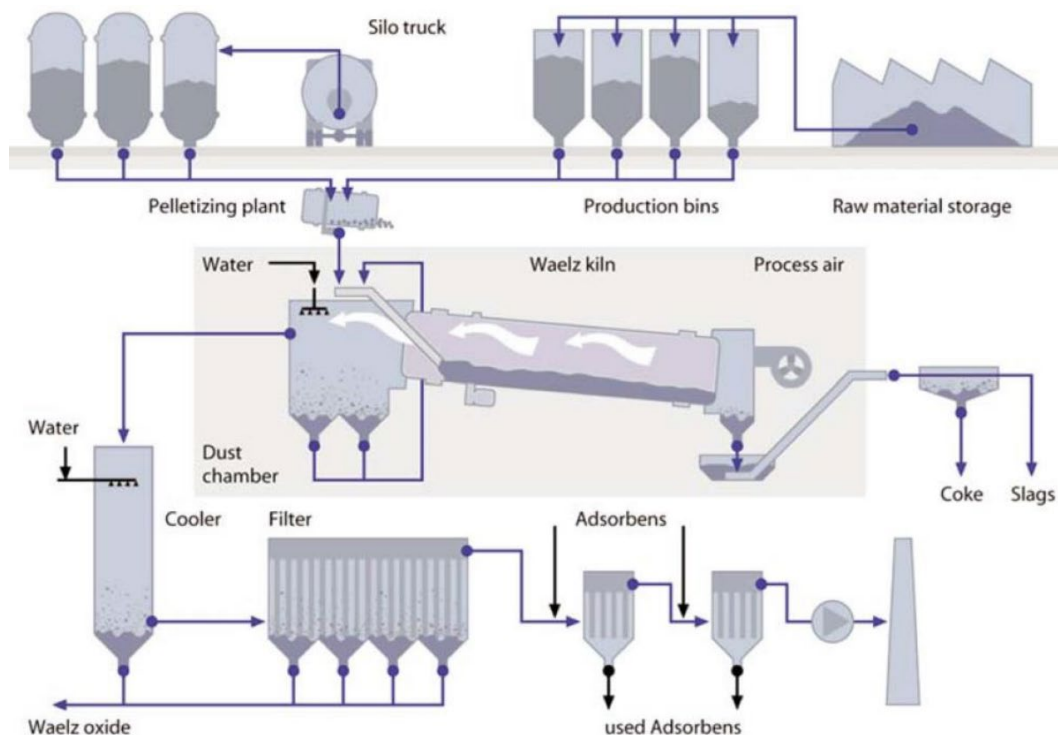
While mechanical methods have a 10–15% lower zinc extraction efficiency compared to pyrometallurgical methods, they are significantly more cost-effective and easier to operate, allowing for mass production.

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#### **2.4.2 Pyrometallurgical methods**

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The dominant process in the field is the Waelz rotary kiln process, widely employed worldwide since the 1970s. This technology is recognized as the best available for the next 25 years (Matthes, Waibel and Keller, 2011). Waelz processes are utilized in the United States, Europe, Southeast Asia (specifically Thailand), and the Middle East (Saudi Arabia). Notably, Russia successfully conducted industrial processing of EAF dust from PAO Severstal using a large-capacity Waelz kiln (over 350 tons per day), demonstrating the effectiveness of the environmentally friendly Waelz process for waste and by-products from ferrous and non-ferrous metallurgy (Bryantseva *et al.*, 2013).



**Figure 5: Waelz process flowsheet (N. Quijorna *et al.*, 2014)**

The process entails the reduction of zinc, cadmium, and lead from pre-agglomerated charge materials, comprising 60% agglomerated feed, 25% coke fines, and 15% SiO<sub>2</sub> flux. These materials are loaded into a rotating tube furnace and maintained at a temperature of 1200 °C for 4 hours (Stovpchenko, Proydak and Kamkina, 2009). Heating the charge results in the removal of zinc vapor and other valuable components via off-gases. Subsequently, upon cooling of the off-gases, the metal vapors condense into fine dust, captured in gas cleaning systems. This dust, known as ‘Waelz oxide’, is crude zinc oxide (50–60% Zn) and is directed towards zinc production, while the slag finds use as a construction material or in road construction (Lytaeva and Pashkevich, 2014). Advantages of the Waelz process include low energy consumption, a well-developed and optimized single-stage process. However, disadvantages encompass the low quality of the product (‘dirty’ zinc oxide), suitable only as a substitute for ore in primary zinc metallurgy, the generation of significant new waste (approximately 700–800 kg per ton of charged dust), and the extraction of only one valuable metal (Antrekowitsch and Rösler, 2015).

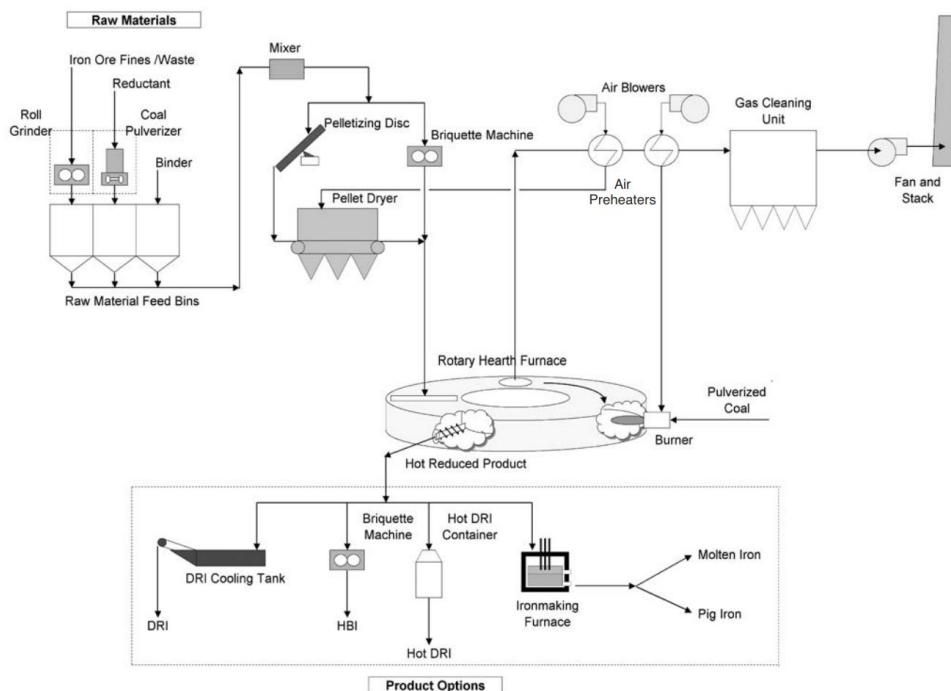
To address the disposal of waste containing high levels of lead (Pb) ranging from 1–2% and zinc (Zn) ranging from 4–7%, companies have successfully resolved this issue by producing metallized briquettes. The technological process involves agglomerating the waste in bowl-type agglomerators, followed by drying and preheating



with off-gases. Subsequently, metallization takes place in rotating kilns at a temperature of 1100 °C. During this process, 99% of lead and zinc are volatilized, along with 85% of alkali metals. The resulting oxides are condensed in the gas discharge system's cold sections, and the dust is collected in the gas cleaning system. The metallized product obtained is utilized in blast furnace smelting, while the fines generated during the reduction process are added to the agglomerated charge (Lin *et al.*, 2017).

When the zinc content in the dust from the rotary kilns ranges from 10–30%, it serves as raw material for non-ferrous metallurgy, whereas metallized briquettes are employed in the blast furnace process. In Japan, six operational plants with capacities ranging from 60,000 to 180,000 tons per year existed. However, since 1977, no new installations of this type have been constructed due to high capital and operating costs (Takaya *et al.*, 2020).

Kobe Steel and Midrex Technologies have developed and implemented the FASTMET and FASTMELT processes for electric arc furnace dust treatment. The FASTMET process was initially introduced in 1995 at a plant in Kakogawa, Japan. It involves direct reduction of iron in Rotary Hearth Furnaces (RHF). The feed materials comprise fine coke as the reductant and briquettes of dust. Operating temperatures range from 1300–1350 °C, with a reduction process duration of 8–10 minutes. The final products obtained are direct reduced iron (with a metallization degree of 75–94%) and crude zinc oxide (50–65% Zn) (Stewart and Barron, 2020).



**Figure 6: FASTMET/FASTMELT process flowsheet (McClelland and Metius, 2003)**

The FASTMELT process, distinct from FASTMET, incorporates an electric furnace. Its primary advantage lies in achieving iron extraction of up to 98%. Economic viability of the technology is realized when processing a minimum of 200,000 tons of dust annually (Stovpchenko, Proydak and Kamkina, 2009). Drawbacks of the process include high consumption of gaseous fuel, approximately 100 m<sup>3</sup> per ton of processed dust, the requirement for a minimum quantity of processed raw materials, inconsistent composition of direct reduced iron, and a low yield of crude zinc oxide.

The PRIMUS Process, developed by the Luxembourg company Paul Wurth, is a pyrometallurgical technology for direct reduction of metallurgical waste. It processes blast furnace and converter sludges, rolling mill scales (including oily ones), and dust from electric steelmaking processes using coal as a reducing agent. The process consists of a two-stage operation with a Multi-Hearth Furnace (MHF) and an electric arc furnace.

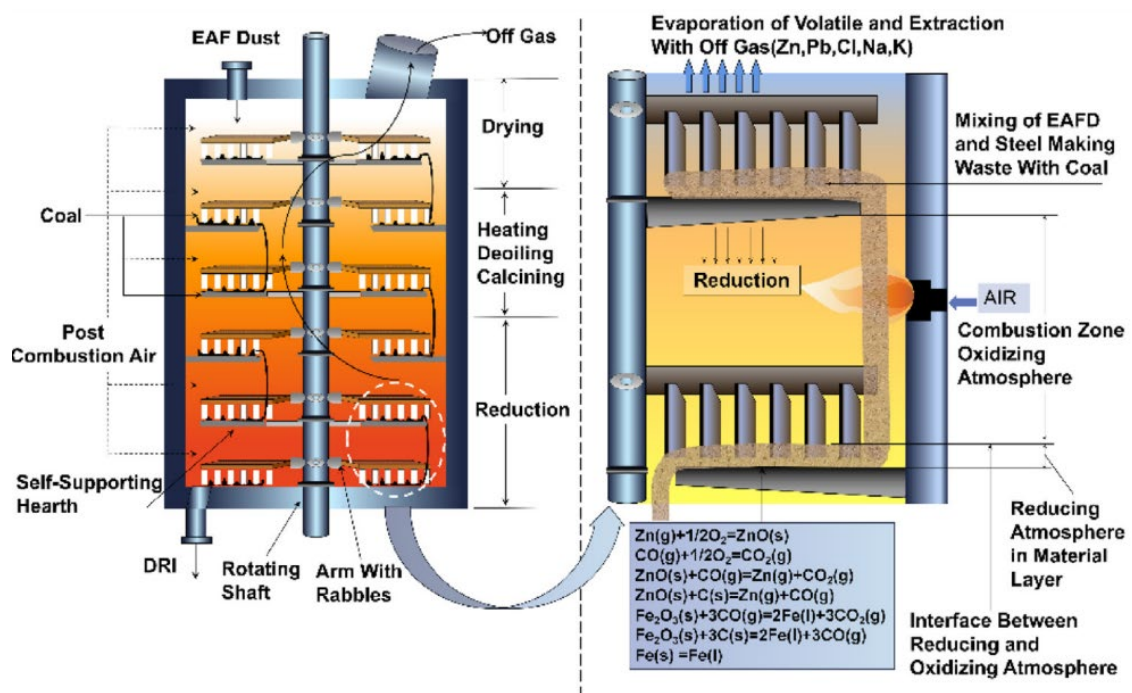
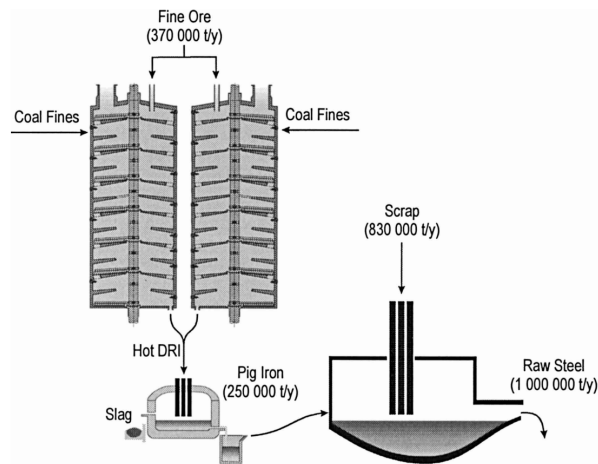


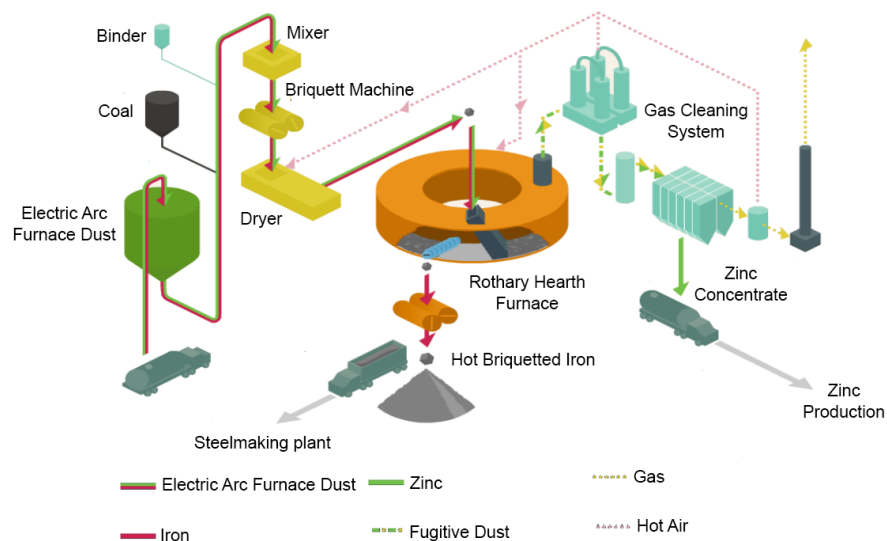
Figure 7: MHF operation (Wang *et al.*, 2021)

The MHF comprises several interconnected chambers stacked vertically. The sludges descend through the chambers, gradually decreasing the concentration of metal oxides in the by-products. Zinc and lead oxides are carried away with the gas and captured in bag filters, while iron oxides are smelted into cast iron in the electric arc furnace. The result is the production of zinc oxide (with over 60% metal content) and granulated cast iron, with the slag used in road construction (Wang *et al.*, 2021).



**Figure 8: PRIMUS process flowsheet (Roth et al., 2001)**

The first PRIMUS installation was established at the Primorec plant in Luxembourg in 2003, with an 85,000-ton capacity. It processes fine particulate dust from electric arc furnaces and rolling mill sludges generated in Luxembourg. The installation includes an eight-hearth MHF and a 10 MW electric arc furnace. It produces 250 kg of cast iron, 350 kg of secondary dust (zinc oxide concentrate), and 200 kg of slag per ton of feedstock, consuming an average of 320 kg of coal and 1.05 MW of electricity. In 2009, a plant was commissioned at the Dragon Steel facility in Taiwan (Alpatova, 2016).

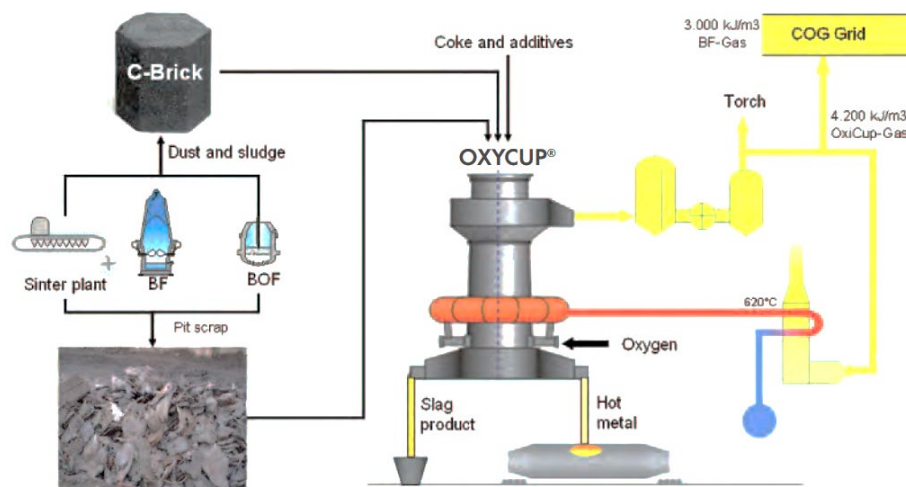


**Figure 9: ZincOx process flowsheet (cp. Baranov et al., 2014)**

ZincOx Resources has developed a technology based on a RHF, similar to FASTMELT. The process involves briquetting iron-containing dust wastes and coal fines with a binder, followed by calcination in the furnace at temperatures above 1250 °C. The coal acts as a reductant, reacting with zinc and iron oxides to produce metallic zinc. At the furnace temperature, zinc vaporizes and reacts with oxygen, forming fine particles of

zinc oxide carried away by the off-gases. Iron remains in the briquette, forming slag. The off-gases are cooled, and solid particles are regenerated in a baghouse filtration chamber. The captured condensate consists mainly of zinc oxide and zinc salts. The technology is highly energy-efficient due to its continuous process and gas combustion in the furnace (Baranov *et al.*, 2014).

Küttner has developed the OXYCUP technology to process metallurgical waste. Fine-dispersed waste materials, including dust and sludge, are mixed with a reductant and formed into C-briquettes. These briquettes, along with coke, fluxes, and other iron-containing waste materials, are loaded into a shaft furnace. The charge is heated from the top and undergoes metal reduction processes at around 1000 °C. The resulting products are continuously discharged as metal (cast iron) and slag. Cast iron with a carbon content of approximately 4% is produced at a temperature of 1500 °C. The evaporated zinc is removed from the furnace space along with the off-gases as a secondary product (Küttner GmbH, 2009).



**Figure 10: OXYCUP process flowsheet (Küttner GmbH, 2009)**

The Waelz technology combined with leaching has shown promising developments and successful implementation by companies like Horsehead (USA), Glencore (Italy), and Akita (Japan) (Kaya, 2023). This technology offers clear advantages, including the production of metallic zinc, reducing reliance on primary zinc smelters, the potential extraction of lead and silver, and the removal of chlorine and fluorine to minimal levels (Antrekowitsch and Rösler, 2015).

However, it is important to consider certain drawbacks, including high investment costs resulting in higher zinc production costs, incomplete iron recovery, and the inability to achieve the required zinc quality during electrolysis.

<b>Parameters</b>	<b>Waelz</b>	<b>RHF</b>	<b>PRIMUS</b>	<b>Oxycup</b>
Zinc content [wt. %]	55–60	50–65	52–60	30–45
Production capacity [kt/year]	25–200	20–300	60–100	250–500
<b>Feed [kg/t]</b>				
Coke fines	200–300	180–250	-	-
Coke	-	-	-	170
Additives	20–200	50	50	200
Coal	-	-	300	160
<b>Products [kg/t]</b>				
Direct reduction iron	-	600–750	-	-
Pig iron	-	-	250–300	300–1000
Raw zinc oxide dust	300–350	50–100	450	50–80
Slag	600–750	-	250	300–400

**Table 3: Comparison of BFS pyrometallurgical processing technologies**

### 2.4.3 Plasma technologies

The application of plasma heating for the extraction of volatile components from dust shows great promise in the field. Plasma technologies offer flexibility in adjusting process parameters and reliable, controllable energy parameters. Additionally, plasma technology has the potential to eliminate the need for a preliminary dust agglomeration stage. The treated dust, after extraction of non-ferrous components, can be reintroduced into the metallurgical cycle.

Several plasma processing technologies are available for zinc- and lead-containing steelmaking dust, such as Tetronics, ScanDust, ArcFume (developed by Mintek, SKF, Davy, Mannesmann-Demag). Examples of plasma-based iron waste processing include the Swedish processes of ScanDust and ArcFume.

ScanDust technology is designed for the treatment of dust from electric arc furnace processes. The dust is mixed with coke and water, excess water is removed, and the mixture is injected into the lower part of the plasma generator. The process produces metal, slag, and gas. The metal is returned to the metallurgical cycle, the slag is utilized in road construction, and the gas can be recycled or used for heat supply. Captured zinc is separated and recovered using other processes (Alpatova, 2016).

The ArcFume process is employed in Hoyanger, Norway, for zinc oxide recovery from dust, with an annual processing capacity of 50,000 tons of dust. However, the

technology faces challenges due to strict requirements for raw material quality, including chemical composition, particle size, and moisture content. In the ArcFume process, dust is mixed with a reductant (coal, coal dust, coke) and fed into a furnace. Zinc oxide is reduced to gaseous metallic zinc, which exits the furnace with the reducing gas. The gas mixture is burned with compressed air above the slag bath. Zinc vapor is converted back to ZnO, while CO and H<sub>2</sub> are oxidized to CO<sub>2</sub> and steam. Fine particles of zinc oxide are carried by the off-gas through a gas cooling system and collected in a bag filter. Iron oxide is reduced to FeO, resulting in a slag predominantly composed of iron oxide, silicon dioxide, and lime (Alpatova, 2016).

Tetronics, a British company, utilizes a plasma arc furnace with a central rotating tilted plasma torch (cathode) and a bath (anode) for hydrometallurgical processing of zinc-containing dusts. This technology employs a single-stage process with one or multiple electrodes, a direct current furnace, and an argon atmosphere. The feed materials, which do not require sintering, consist of powdered waste, flux, and coke. Reduction occurs at temperatures ranging from 1500 to 1550 °C. Organic and inorganic compound decomposition takes place primarily at 1500 °C within the reactor, with the plasma arc core reaching temperatures of approximately 10,000 °C. The resulting products include cast iron, slag, and crude zinc oxide. Manufacturers have reported the following advantages of this technology as minimal environmental impact, easy control and maintenance, relatively low capital and operating costs and versatility in processing various waste materials. Tetronics' plasma systems are currently operational in Japan, the United Kingdom, Italy, Germany, and Korea (Toporkova *et al.*, 2021).

Mintek (USA) has developed a plasma arc processing technology for electric arc furnace dust. This technology incorporates a plasma arc complex consisting of four furnaces, with power capacities ranging from 30 kW to 1 MW. The furnaces operate using direct current, with a graphite electrode serving as the cathode and the furnace bath melt acting as the anode. The process throughput varies from 5 to 100 kg/h for a 200 kVA furnace. The plasma-forming gas, either nitrogen or argon, is supplied through the central hole in the graphite electrode (Toporkova *et al.*, 2021).

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#### **2.4.4 Liquid-phase iron recovery**

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Since the early 1970s, there has been significant global development of liquid-phase iron recovery processes for dispersed secondary raw materials, including blast furnace and converter dusts, sludges, and mill scale. These processes aim to produce cost-

effective cast iron from waste materials. Notable processes include COREX (Austria, Germany, since 1977), ROMELT (Russia, 1979), HIs melt (Germany, 1984), DIOS (Japan, 1988), AusIron (Austria, 1994), and FINEX (Austria) (Alpatova, 2016). Liquid-phase processes often suffer from the disadvantage of being challenging to implement on an industrial scale and requiring substantial capital investment.

Typically, the liquid-phase process consists of two stages. In the first stage, the raw material undergoes heating and partial reduction (up to 50%) using gas generated in the second stage, where final reduction of the product occurs. Preliminary reduction is performed in shaft furnaces or fluidized-bed reactors. Final reduction takes place in low-shaft furnaces (including electric furnaces) or converter-type units. The process involves preliminary reduction of materials in a shaft furnace (similar to the Midrex furnace with hot discharge) using gas generated in the gasifier. The pre-reduced material is then loaded for final reduction, along with coal gasification facilitated by oxygen injection into the unit (Li *et al.*, 2019).

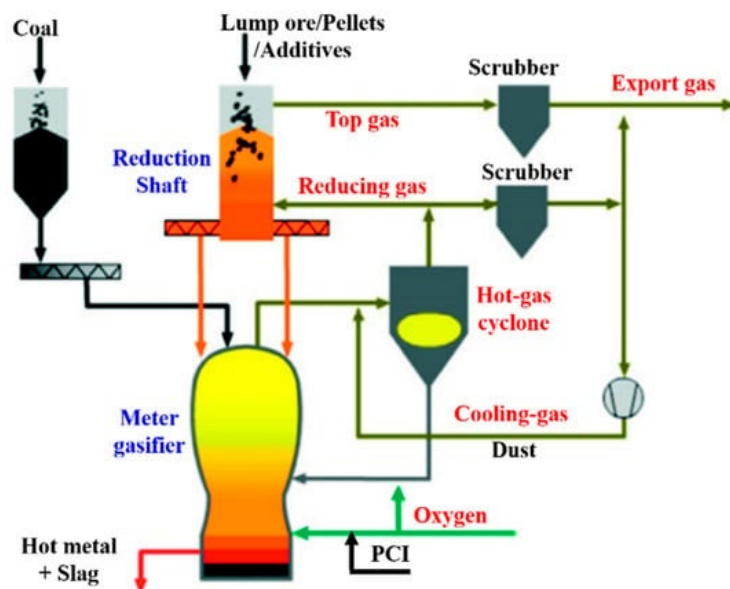


Figure 11: COREX process flowsheet (Li *et al.*, 2019)

The most established liquid-phase process is the COREX process, jointly developed by Voest Alpine (Austria) and Korf Engineering (Germany). Operational installations currently include capacities of 60,000 tons/year (Germany) and 300,000 tons/year (ISCOR, South Africa). A second-generation COREX plant with a capacity of 600,000 tons/year has been developed (Alpatova, 2016).

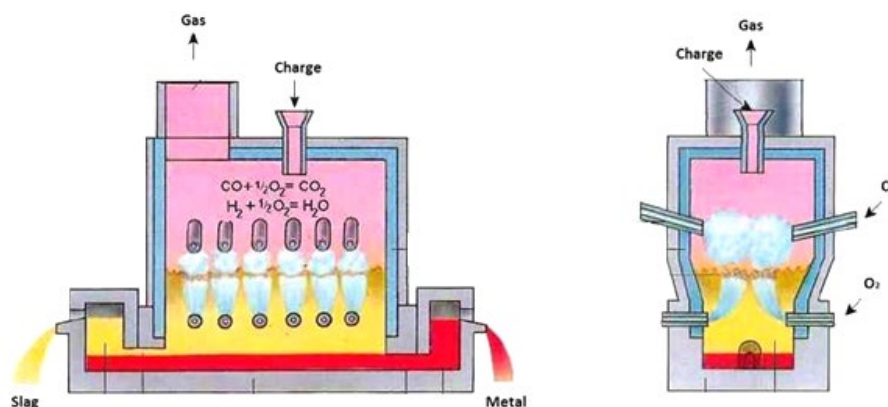
The FINEX process, a development based on the COREX process, was jointly developed by Voest Alpine and the research center of POSCO. It employs fine-sized



iron ore feed, with particle sizes up to 8 mm. In the FINEX process, pre-reduction of the ore occurs in fluidized-bed reactors, followed by the production of pig iron from partially metallized feed in a furnace-gasifier. The pre-reduced feed, with a metallization degree of 85–90%, is briquetted and introduced into the furnace-gasifier via loading hoppers. The processes within the furnace-gasifier of the FINEX unit are similar to those in the COREX process (Yi *et al.*, 2019).

The development and research program for this technology involved retrofitting a 600,000-ton/year COREX plant into a FINEX module, testing and refining the technology, and subsequently constructing a new module with higher capacity. The second FINEX unit, with a pig iron production capacity of 1.5 million tons per year, was launched in April 2007 at a plant located in South Korea (Kurunov, 2015).

The ROMELT technology, initially implemented in 1984 at Novolipetsk Steel and later tested in a pilot plant in Daejeon, Republic of Korea, has been further developed and applied on an industrial scale. Notably, in 2008, an industrial plant with a capacity of 32,000 tons of pig iron per year was established in Balkhash, Kazakhstan, followed by a plant with a capacity of 205,000 tons of pig iron per year in Myanmar in 2015. ROMELT is a continuous method utilizing energy coals to produce pig iron from various iron-bearing raw materials and waste. It involves blowing an oxygen-air mixture into the furnace, stirring the molten slag, and continuously loading iron-bearing raw materials and coal onto the surface of the slag melt. The resulting reduced iron is collected at the bottom of the furnace, while the metal and slag are discharged through different levels. The volatile elements carried away by flue gases are precipitated in dust, which can be used to obtain non-ferrous metals. Despite the establishment of a ROMELT plant in Myanmar, the technology has not gained widespread industrial adoption (Kleiman, 2004).



**Figure 12: Schematic views of the inner core of ROMELT stove (Polyanskiy D., 2012)**



The Dios process was initially implemented on a semi-industrial plant at the NKK Keihin plant in Japan in 1993, with a nominal capacity of 180,000 tons of pig iron per year. The process consists of three stages: charge preheating, preliminary reduction in a fluidized bed reactor, and final reduction in a liquid-phase reduction reactor. The charge, composed of fine ore and powdered waste, is preheated to 600 °C in a pseudo-liquefied layer reactor. It undergoes preliminary reduction at a temperature of 780 °C with a degree of reduction ranging from 27% to 30% in the fluidized bed reactor. Finally, the charge goes through final reduction in the liquid-phase reduction reactor under a pressure of  $2 \times 10^5$  Pa (Kitagawa, T, Iwasaki, K, Kojima, O and Sekiguchi, 1999).

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#### 2.4.5 Hydrometallurgical methods

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##### Acid leaching

Initially, research focused on developing sulfuric acid-based technologies for treating metallurgical dust. However, despite extensive research, industrial implementation of sulfuric acid leaching has not been successful (Vereš, Jakabský and Lovás, 2011).

(Oustadakis *et al.*, 2010) and (Havlik *et al.*, 2012) have demonstrated that developed sulfuric acid leaching technologies yield low zinc extraction rates from dust with high iron content (over 45%) and zinc content (over 8%). When leaching such dust with approximately 60% sulfuric acid at stoichiometric zinc concentration, only 40% reacts with zinc, while the remaining 60% reacts with iron. The presence of impurities like calcium oxide or magnesium oxide in the dust further reduces the acid's zinc-reactive capacity to 10–12%. Increasing the acid concentration exacerbates the issue by increasing the reaction with iron (Kekki, Aromaa and Forsén, 2012). Consequently, this necessitates significant expenses for impurity purification prior to zinc electrolysis and additional costs for environmentally safe disposal of these precipitated impurities.

Leaching with low-concentration acid solutions (0.1 to 2.0 M) at temperatures ranging from 18 to 60 °C presents a potential solution to the problem. Oustadakis *et al.* (2010) presents research on this topic, indicating that the reaction rate for zinc remains relatively constant within the investigated acid concentration range, unlike the reaction rate for iron. The study achieved a 90% zinc extraction and less than 50% iron extraction into the solution.

However, a significant drawback of this method is the substantial dilution of the technology. Processing 1 ton of dust can yield approximately 20–25 tons of solutions.

Although semi-industrial tests have been conducted, industrial application is yet to be realized.

Vereš, Jakabský and Lovás (2011) describe research on zinc extraction using acid leaching with the application of microwave heating for sulfuric, hydrochloric, and nitric acids. This method shows a slight increase in extraction (1.5–2% compared to traditional sulfuric acid leaching methods). According to the data presented, sulfuric acid exhibits the highest degree of zinc extraction.

In the study conducted by Havlik, Friedrich and Stopic (2004), the leaching of dust from a steel foundry plant in the USA (26% Zn, 40% Fe) was investigated using sulfuric acid under atmospheric and elevated pressure conditions. The results showed approximately equal zinc leaching efficiency. The zinc extraction ranged from 32% (with sulfuric acid solution concentration of 0.2 M) to 76% (sulfuric acid concentration of 2 M) using an autoclave under leaching conditions of 2 hours at 270 °C, and from 53% (0.2 M) to 79% (2 M) using atmospheric leaching for 4 hours at 70 °C. When high concentration sulfuric acid (4 M) was employed, zinc extraction reached 97%, but nearly all the iron transferred into the solution.

There exists a technology based on the dissolution of ZnO and ZnFe<sub>2</sub>O<sub>4</sub> in hydrochloric acid, obtained from iron salts (Langová, Leško and Matýsek, 2009). The dust was leached in an autoclave with stirring using a solution composed of FeCl<sub>3</sub>, FeCl<sub>2</sub>, and HCl, obtained by reacting metallurgical waste with gaseous chlorine. Oxygen is also supplied to the autoclave. As a result, FeCl<sub>2</sub> is oxidized to FeCl<sub>3</sub>, which extensively hydrolyzes at 175 °C and produces HCl. The resulting hydrochloric acid reacts with ZnO and ZnFe<sub>2</sub>O<sub>4</sub>, which are the main zinc-containing phases in the dust. Residual FeCl<sub>3</sub> also reacts with ZnO. According to the data presented in these studies, zinc extraction of 80–85% is achieved, while Fe<sub>2</sub>O<sub>3</sub> remains predominantly insoluble in the dust. Despite the high extraction rates, this technology has not found industrial application due to the requirement of large quantities of pure chlorine for the production of leaching solvents, which is costly and environmentally hazardous.

Elevated halogen content in technogenic deposits poses challenges during electrolysis or precipitation methods in both sulfuric acid leaching and other techniques used. This issue affects certain industrial sites.

Another branch of acid leaching methods involves the use of organic acids like acetic acid (Dreisinger, Peters and Morgan, 1990), (Barrett, Nenniger and Dziewinski, 1992)

or triphenylphosphite (Baba and Adekola, 2008). Pre-treatment of the dust is performed to eliminate chlorides, as even small concentrations of chlorine hinder the process by transferring iron into the solution during leaching. Lime and non-ferrous metal oxides dissolve, forming acetates that enter the solution. The residue mainly consists of iron oxides (40–60%) and insoluble impurities like silicon oxide. Depending on the initial dust composition, the residue may contain up to 4–5% zinc. However, this residue can be directed for further processing in blast furnaces, as the presence of silicon oxide does not affect the process and even reduces the required flux. The zinc content can be reduced by diluting this dust with iron ore (Barrett, Nenniger and Dziewinski, 1992).

The solution containing metal acetates and calcium undergoes metal precipitation by adding hydrogen sulfide acid, which forms insoluble sulfides of the target components. The precipitate is sent to a zinc production plant, while the solution is purified to remove calcium. Sulfuric acid is added for calcium precipitation, resulting in the formation and precipitation of calcium sulfate. After calcium precipitation, if necessary, the solution undergoes further purification to remove metal cations before being returned to the main process. Several plants in the USA were based on this technology, but they were shut down due to low profitability. The zinc extraction efficiency using this method is approximately 80%. However, when processing raw materials with high zinc content, issues may arise with the leaching residue, as the zinc content remains significant. Additionally, the resulting zinc product contains a high level of impurities, reducing its value and overall profitability (Dreisinger, Peters and Morgan, 1990).

Another technology utilizing citric acid for zinc leaching is described by Larba *et al.*, (2013), but it has not undergone industrial testing. One advantage of this method is the potential for selective zinc extraction. However, the extraction efficiency is considerably lower, achieving less than 70% zinc extraction from pure zinc oxide. Extraction from ferritic forms or other refractory compounds would be even lower. Moreover, a drawback of this technology is the high degree of required dilution, with a recommended liquid-to-solid ratio (L/S ratio) of 20:1.

### Alkaline methods

Alkaline methods are more selective but are also more complex and less efficient in zinc extraction into solution. The use of alkaline solutions also has other drawbacks: the high process temperature requires expensive autoclave equipment, and the high

cost of reagents necessitates the creation of conditions for their maximum regeneration (Mordoğan, Cicek and Işık, 1999).

However, numerous studies related to alkaline leaching processes demonstrate that alkaline leaching is an energy- and resource-saving method compared to acid leaching. For instance, (Mordoğan, Cicek and Işık, 1999) describe method for extracting zinc from metallurgical dust using sodium hydroxide leaching under harsh conditions (6–8 mol/L NaOH; above 200 °C). Sodium hydroxide has been used as a reagent for processing dust due to the insolubility of iron in alkaline solutions, rendering some other dust components inactive in the leaching process (Farnasov *et al.*, 2016). Consequently, the leach solutions primarily consist of zincate ions, simplifying subsequent purification processes.

Zinc extraction depends on the mass ratio of sodium hydroxide to dust and the ferrite structure (Youcai and Stanforth, 2000). In a study conducted by Li *et al.* (2012), leaching was performed under various conditions, and it was determined that ultrasonic mixing does not impact zinc extraction significantly. Leaching in a 6 M sodium hydroxide solution at 90 °C for 4 hours achieves approximately 74% zinc extraction. Larba *et al.* (2013) described optimal conditions were identified as follows: a temperature of 95 °C, L/S ratio of 7:1, 10 M NaOH, and a leaching duration of 2 hours. Under these conditions, 85% of the zinc was extracted.

According to Aromaa *et al.* (2013), zinc present in the raw material in oxidized form can be efficiently leached in an alkaline solution (up to 85% of zinc and no more than 1% of iron dissolve in the solution). However, zinc in the form of ZnS and ZnFe<sub>2</sub>O<sub>4</sub> hardly dissolves (Jiang, Guo and Zhao, 2010).

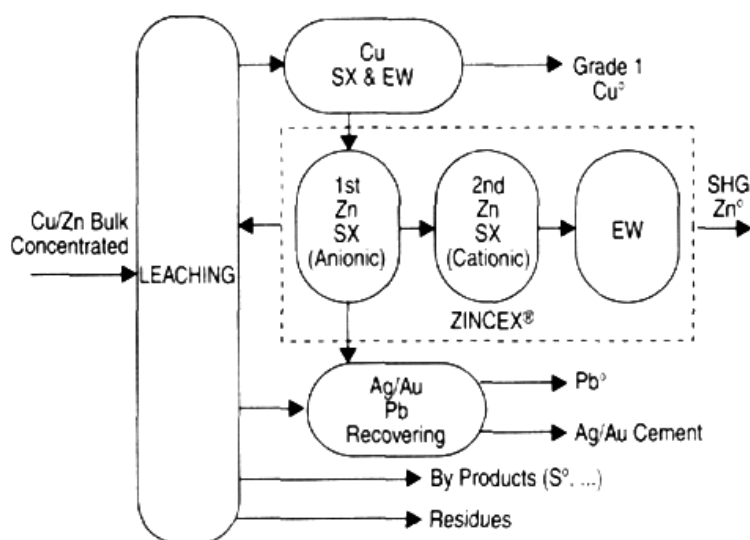
There are also combined methods, not yet implemented in industry, such as a two-stage technology involving co-roasting the dust with soda followed by alkaline leaching. This method enhances the extraction of zinc from oxide and sulfide, but it barely affects the extraction of zinc from ferrite (Xia and Pickles, 1999).

The ammoniacal method (ammonia-ammonium extraction of metals from refractory ores and technogenic raw materials) is one of the promising leaching methods. The method of ammonia leaching can be applied to metals (such as copper, nickel, and zinc) capable of forming ammonia complexes. The main drawback of ammonia leaching is the presence of zinc ferrite, which is insoluble in an ammonia environment. Therefore, for electric arc furnace dust, the extraction of zinc into the solution does not

exceed 30–40%. When using ammonium sulfate, zinc extraction reaches 85% under the conditions of a temperature of 90 °C, a leaching time of 4 hours, and a reagent concentration of 4 mol/dm<sup>3</sup> (Halli *et al.*, 2018).

### Industrial Application

The ZINCEX technology enables the processing of primary and secondary zinc raw materials and the extraction of zinc from a chloride medium in the presence of significant impurities. The key stage of the process is liquid extraction, used for concentrating and purifying the zinc solution, from which metallic zinc, zinc sulfate, zinc oxide, and other chemical compounds can be obtained.



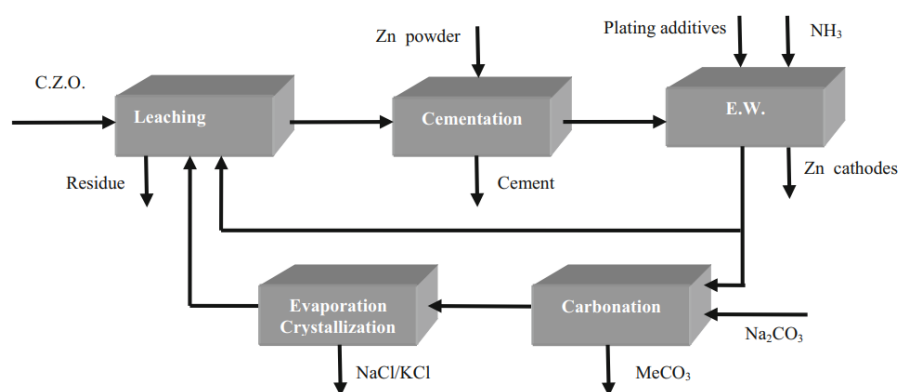
**Figure 13: Conceptual diagram of ZINCEX process applied on bulk concentrate (Diaz *et al.*, 2001)**

According to this schematic diagram, secondary zinc materials with a sufficiently high zinc content are successfully processed. However, the application of the ZINCEX technology to BFS may not be economically feasible.

The technology includes the following operations: leaching, extraction, washing, re-extraction, and regeneration stages. Zinc raw materials are leached in a sulfuric acid, hydrochloric acid, or ammonium chloride solution. Depending on the raw material, atmospheric leaching, heap leaching, pressure leaching in an autoclave, or bioleaching can be employed. To extract zinc from the aqueous phase into the organic phase, the rich solution comes into contact with an organic solvent in the first stage. Zinc from the washed organic extract is re-extracted with an acid solution, yielding a very pure zinc sulfate solution suitable for obtaining SHG Zn, ZnSO<sub>4</sub>, and ZnO (Diaz *et al.*, 2001).

This technology underwent successful trials in industrial plants and found application in the Scorpion project in Namibia. It has also been successfully implemented in Spain for battery recycling (Garcia *et al.*, 2000).

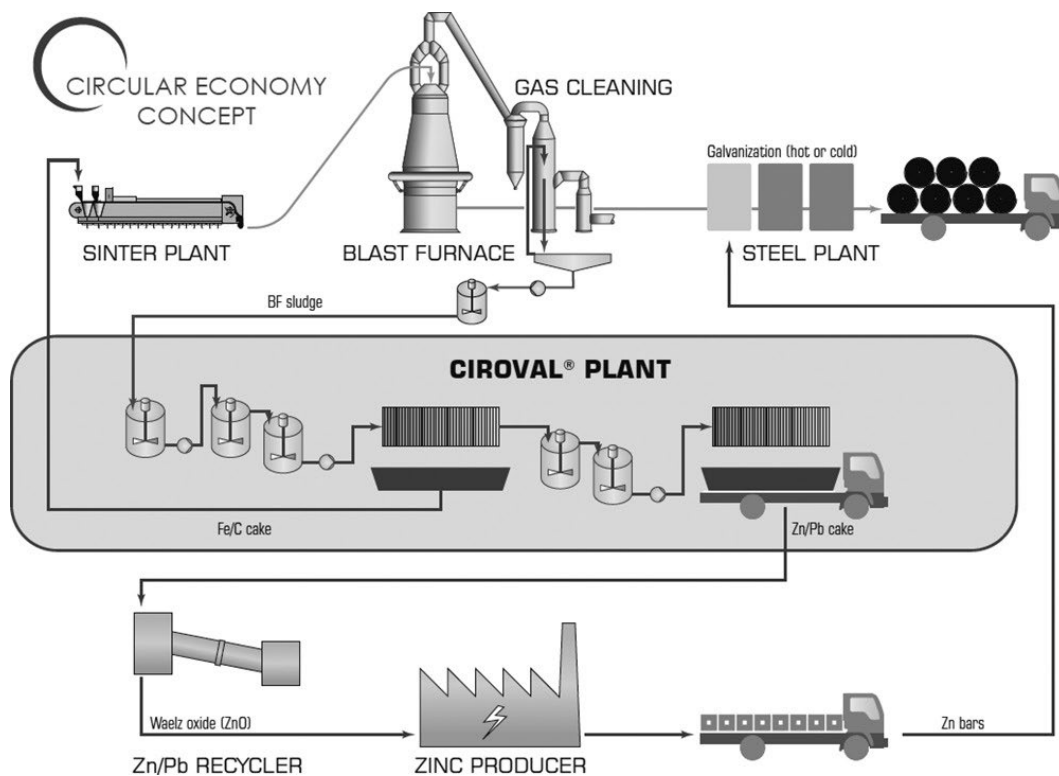
The EZINEX technology enables the processing of various zinc-containing materials, such as dust from steel-making processes and blast furnaces, leaching residues from the Waelz process, and others. The technology involves the following main operations: leaching of the dust using ammonium chloride, purification of the solution from impurities, zinc electro-deposition, and purification of the solution from alkali metal chlorides (Maccagni, 2016).



**Figure 14: EZINEX process block diagram (Maccagni, 2016)**

The zinc-containing oxides present in the dust dissolve into the solution, while zinc ferrite and iron oxides remain in the residue. The purified electrolyte, after filtration, is directed to electrolysis with a titanium cathode and a graphite anode. The emission of hazardous chlorine gas during the electrolysis process is eliminated through constant control of the technological process. Since ammonia is consumed during this process, its addition is necessary to maintain the electrolyte pH at a level of 6.0–6.5 (Olper, 1997).

CIROVAL, developed by Paul Wurth, a subsidiary of the SMS group, is an eco-friendly hydrometallurgical process designed for the efficient utilization of fresh BFS. CIROVAL comprises two stages: low-temperature treatment and selective oxidizing leaching. Its objective is to recycle iron-carbon by-product with zinc content below 0.3% and lead content below 0.1%. The process involves selectively leaching zinc and lead under acidic and oxidizing conditions in Step 1, followed by neutralizing the filtrate in Step 2 to precipitate heavy metals as hydroxide cake (see Figure 15).



**Figure 15: CIROVAL process flowsheet (Ante *et al.*, 2019)**

The CIROVAL process converts BF sludge into three valuable products. Firstly, purified carbon-iron products, free from zinc and lead, can be used as secondary raw materials in sinter plants. Secondly, the zinc-lead concentrate, containing various heavy metals, can be utilized as secondary raw materials in the non-ferrous industry. Finally, the process generates a byproduct of salty water, which can be recycled within the iron and steelmaking plant or concentrated for use in the salt industry.

The development of the CIROVAL process commenced in 2013 with laboratory experiments using BFS from European plants. Promising results led to the design of a mobile pilot plant unit capable of treating up to 150 kg of BF sludge per batch. Test campaigns were conducted at ArcelorMittal Dunkirk in the first half of 2016, demonstrating the ability to reduce zinc and lead content. This success prompted the industrial-scale implementation of the CIROVAL process at the ArcelorMittal Dunkirk facility. The commissioning of the first operational CIROVAL plant is expected by the end of 2020 (Ante *et al.*, 2019).

## 2.5 Blast Furnace Sludges at Severstal's

Cherepovets Steel Mill (CSM) – one of the largest enterprises in the metallurgical industry of Russia, is a part of the Severstal company. It is located in the city of Cherepovets, Vologda Oblast. The plant was founded in 1955 and has since

continuously expanded and modernized. CMP encompasses all the major processes of metallurgical production, from raw material extraction to steel smelting and the production of finished goods. It manufactures a wide range of products, including hot-rolled and cold-rolled steel, galvanized and zinc-coated sheets, structural and shaped profiles, as well as pipes for various applications. CMP is a key supplier of steel to many sectors of the Russian economy, including construction, automotive industry, shipbuilding, and mechanical engineering.

However, like any metallurgical plant, CMP faces the challenge of managing production waste, including blast furnace slag. Due to its high zinc content, the waste is not suitable for recycling in the blast furnace process and therefore needs to be stored in a slag landfill.

Type of waste	2019	2020	2021	2022
Slag	4.27	4.17	4.50	4.32
Scrap metal	0.83	0.80	0.86	0.80
Process waste	0.39	0.27	0.29	0.27
Scale	0.33	0.33	0.32	0.29
Aspiration and gas cleaning dust	0.21	0.23	0.28	0.32
Steelmaking sludge	0.18	0.19	0.32	0.29
CSM main waste	6.21	5.99	6.57	6.29

**Table 4: Main types of waste generated by CSM, million tonnes (Severstal, 2022, 2023)**

### 2.5.1 Sludge storage at PAO Severstal

Sludge storage facility No. 2 is designed to store dried zinc-containing sludge from blast furnace circulating water supply and gas cleaning system. The sludge can be further utilized as a secondary resource in production. The gas cleaning sludge is classified as low-hazard, belonging to Hazard Class 4. The annual inflow volume is 37,800 tons, and the sludge will be stored for a period of 20 years. The landfill area, along with associated facilities, covers 7.2 hectares.

The storage facility, owned by PAO Severstal, is situated north of the main industrial site. Specialized MAZ trucks with a payload capacity of 20 tons are used for transporting the sludge to prevent any waste leakage into the surrounding environment. The storage technology involves a vertical stacking scheme with three stages, each divided into four sections. The stacking process begins from the furthest section and progresses upwards using a front loader and bulldozer. Compaction is then carried out using the bulldozer.





**Figure 16: View of the sludge storage facility No. 2 (Yevgeny Vinogradov, 2021)**

The landfill site consists of two zones: the production zone and the utility zone. The production zone is designated for direct sludge storage and is enclosed by a 4-meter-high dam made of blast furnace slag. This dam acts as a barrier and ensures the stability of the sludge masses. To prevent unauthorized access, the perimeter of the site is enclosed by a fence. Additionally, three monitoring wells are installed for groundwater monitoring purposes.



**Figure 17: Stockpiling of BFS at storage facility (Yevgeny Vinogradov, 2021)**

The utility zone houses various facilities, including a dispatcher's office, a utility building, and a bio-toilet cabin. Furthermore, the site includes a vehicle parking platform with a canopy that accommodates four parking spaces, a refueling area, a wheel washing area, and a reservoir for collecting rainwater and meltwater.



**Figure 18: 3D model of a zinc-containing blast furnace sludge storage site project (OOO Energostal, 2016)**

The storage facility site does not possess a fertile soil layer, and there is no requirement for additional land diversion or acquisition. During positive temperatures, the site surface is irrigated with technical water using a sprinkler machine, achieving an estimated effectiveness of 85%. To mitigate dust emission within the site, a temporary access road with a turning area is constructed using relocatable reinforced concrete slabs. The enclosing dam serves a protective function in the initial stage. The site's contribution to air pollution is minimal, with no violations of hygienic air quality standards at the sanitary protection boundary. The landfill site is included in the project for maximum permissible emissions of pollutants from the enterprise.

There are no existing water networks within the site. Bottled water is provided for drinking and domestic purposes. A bio-toilet serves as a collector for domestic wastewater. The project incorporates two drainage ditches, an inner and an outer one, to manage rainwater and meltwater runoff. Collected rainwater and meltwater are directed to a settling tank, where suspended solids are removed through treatment. The water from the settling tank is used for equipment wheel washing and site irrigation. Some of the water is also transported to CSM to supplement the circulating water supply of gas cleaning unit No. 1—5. Drainage water is transported via tanker trucks with a capacity of 17 m<sup>3</sup>, as there is no sewage system on the site. Moreover, this water is utilized for sludge irrigation up to a depth of 14 cm, establishing a closed-loop system without any discharge into water bodies. This demonstrates the efficient utilization of water resources at the site.

To safeguard groundwater, an anti-filtration screen (geomembrane) made of geosynthetic materials (polyethylene film) is installed at the bottom of the excavation pit and on the inner slopes of the enclosing dam.

According to SanPiN 2.2.1.1.1200-03 regulations, the prescribed size of the regulatory sanitary protection zone for the site is 500 meters. This is integrated into the overall regulatory sanitary protection zone of the entire enterprise, which covers 1 km.

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### **2.5.2 Recycling of blast furnace sludge at Severstal**

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To mitigate the anthropogenic impact on soil and land degradation, as well as to preserve the capacity of the existing blast furnace slag storage area and reduce dust emissions during waste disposal, Severstal has devised multiple sludge recycling solutions.

The utilization of BFS as an iron-containing additive in cement clinker production has seen significant growth. The quantity of sludge shipped in 2021 reached 115,000 tons, a notable increase compared to 2,700 tons in 2020. This initiative has led to a waste recycling rate increase from 95% in 2020 to 96.6% in the first half of 2021 for Severstal. Waste disposal was reduced by 82,000 tons, resulting in improved environmental conditions. Moreover, the project generated an economic benefit of 279.8 million rubles for the company in 2021 (Severstal, 2022). In 2022, 34,000 tonnes were recovered in-house and shipped to consumers (Severstal, 2023).

Additionally, the CSM has developed the production of sludge coal briquettes for steelmaking purposes. These briquettes are created using a rigid vacuum extrusion method and consist of a mixture comprising BFS (30%), dehydrated sludge area scale (30%), and dust from gas cleaning of electric arc furnace production (40%). This composition enables the production of self-reducing briquettes, as the carbon content in the BFS facilitates iron reduction during melting under converter smelting conditions. A continuous cost reduction of 64.8 million rubles is expected over a period of 10 months (Severstal, 2022).

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## **2.6 Discussion and selection an optimal processing method**

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Literature analysis indicates that for steelmaking dust with high zinc content, both pyrometallurgical and hydrometallurgical approaches are considered for extraction. Among the pyrometallurgical methods proposed, a significant group focuses on reducing zinc oxide to metal using various reagents, followed by distillation. These methods differ slightly in terms of equipment used.

However, pyrometallurgical processing methods share common drawbacks. The resulting product is zinc in oxide form with high impurity content, essentially becoming secondary dust. The reduction process requires substantial consumption of coal, coke fines, or other reducing agents. The released zinc negatively affects furnace lining lifespan. Processing zinc-containing products in zinc production plants incurs additional material and energy costs, impacting the cost of refined zinc.

Hydrometallurgical processing methods, mainly acid or alkaline leaching variants, offer undeniable advantages. They enable comprehensive processing of raw materials, achieving high extraction of all valuable components, high economic efficiency, reduced atmospheric pollution from harmful emissions, and improved working conditions. However, their application is hindered by the fact that zinc in the dust exists as practically insoluble ferritic compounds in alkaline solutions, while acid usage results in iron dissolution.

An effective solution to the challenge of efficient hydrometallurgical processing of zinc-containing dust can be the application of alternative solvents, such as salt solutions (e.g., ammonium chloride). The use of such solvents allows for maximum zinc extraction into the solution while preserving iron quantitatively in an insoluble residue.

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## 3 Experimental investigation

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### 3.1 Methods

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Samples of blast furnace slag were collected from CSM's sludge storage facility No. 2. The sample collection was based on 12 selections at different areas and depths of the sludge accumulator, followed by mixing. Each material sample was dried at 90 °C and milled to a particle size of 0.5 mm.

Mineralogical analysis, including X-ray phase, optical, and microprobe methods, was conducted at the Research Mineralogical Laboratory in ZAO IVS. Optical examination was carried out using the Olympus BX51 polarizing microscope equipped with a SIMAGIS 2P-3C digital camera. Microprobe analysis was performed using an analytical complex based on the VEGA 3LMU scanning electron microscope with an energy-dispersive microanalysis and wave-dispersive microanalysis system. Polished sections were prepared from sludge samples for this analysis.

Powder preparations were prepared to identify the main mineral phases. They were evaluated on the D2 PHASER portable X-ray diffractometer (CuK $\alpha$ ) in the range of 2 $\theta$  angles from 5 to 90°. Diffractograms were processed in the DIFFRAC.EVA program, with qualitative X-ray phase analysis compared to ICDD PDF 2 standards.

Granulometric analysis employed the Mechanobr-Techika ASV-300 vibratory sieve analyzer with 0.01, 0.071 and 0.045 mm sieves. The dispersion continued for 30 minutes, the obtained size classes were weighed and placed in individual envelopes. Selected samples were then sent for chemical analysis.

Chemical analysis took place at the Gipronickel Institute, St. Petersburg. To determine the content of various elements such as iron, copper, nickel, zinc, lead, manganese, aluminum, chrome, magnesium, potassium, and silicon oxide, the iCAP 7600 Radical atomic emission spectrometer was used. The mass fraction of fluorine was determined by ionometry on the T70 automatic titrator. The determination of the mass fraction of sulfur and carbon was measured by infrared spectrometry on the SC144 gas analyzer.

To examine the applicability of gravity methods for processing BFS, wet sieving with 0.045 mm cell size of sample into two size classes was performed. Additionally, the feasibility of wet magnetic separation was assessed with Mechanobr-Techika EBM-32/20 drum-type magnetic separator. The resulting products

were dried at 105 °C and their concentration ratio were calculated. Selected samples were analyzed for Zn, Fe, C, and Ca.

The most promising solvent reagents, namely  $\text{NH}_3 + \text{NH}_4\text{Cl}$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{HCl}$ , were chosen following a thorough literature review and an analysis of industrial experience.

Ammonium chloride leaching was conducted in an autoclave with mechanical stirring to minimize ammonia loss. Raw material and reagents were loaded into the autoclave, then heated with constant stirring to the specified temperature (vapor pressure at 80 °C was 2–5 bar).

For acid leaching, an acid solution was prepared in a glass reactor. Once it reached the target temperature, the test material was introduced into the reactor, prompting the initiation of time tracking and agitation (see Figure 19). A stable pulp level was ensured during the leaching process.



**Figure 19: Leaching setup**

After the set time, the pulp was filtered. A sample was extracted from the filtrate for quantitative analysis of the dissolved substances. The cake was washed until neutralization, employing a minimum of three equal water volumes, considering L/S of 2 m<sup>3</sup>/t (see Figure 20). The rinsed material was dried at 105 °C until a consistent mass was obtained. Then it was then weighed, pulverized, partitioned, and a portion was chosen for further analysis.





Figure 20: Cake washing

### 3.2 Characterisation of the blast furnace sludge sample

Evaluating the potential of BFS recycling requires identifying its chemical and mineralogical composition and the zinc's form in the dust. Knowing the minerals' quantity and form helps to set optimal experimental conditions.

The sludge consists of zinc, iron, and manganese oxides, spinel, metallic iron, and graphite mineral phases. Manganese and quartz oxides are minor constituents. Figure 21 shows a diffractogram of the analyzed sample.

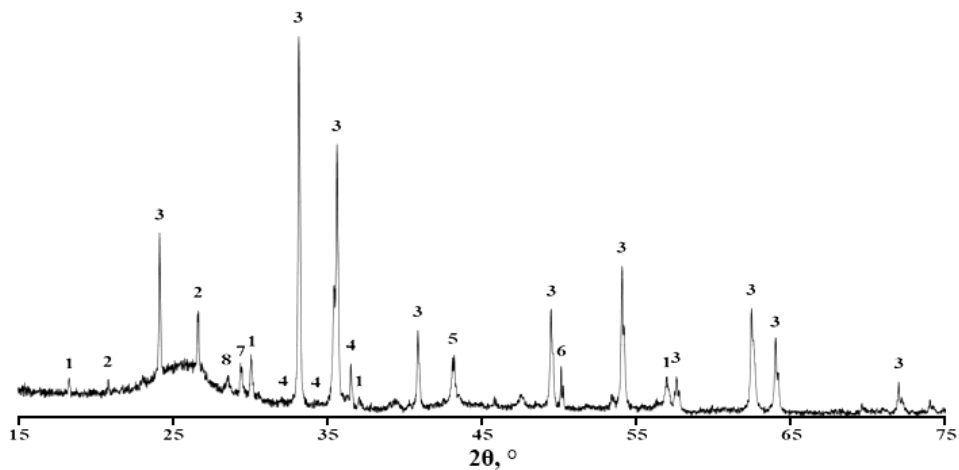
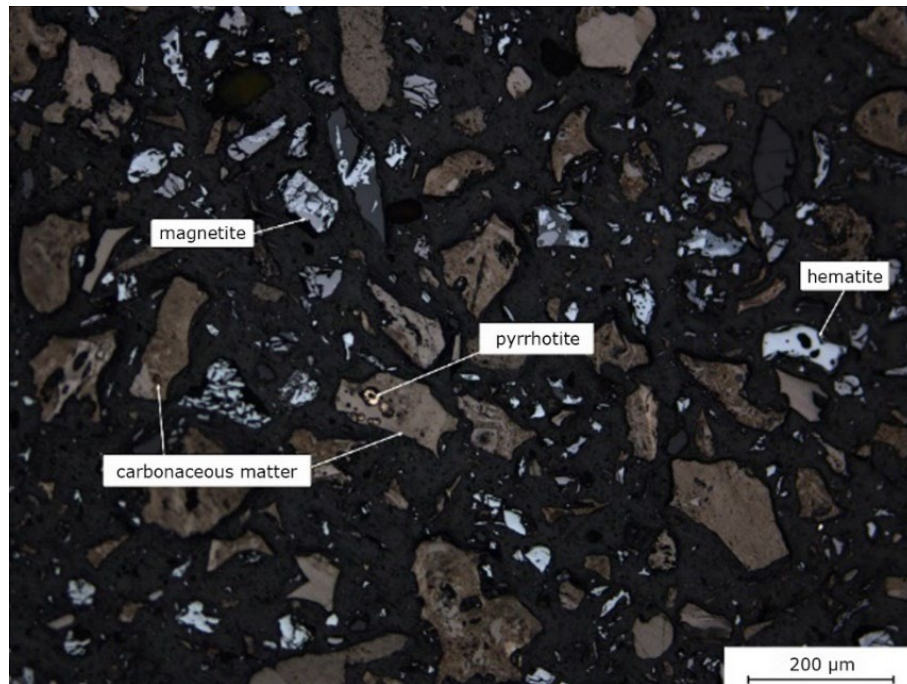


Figure 21: X-Ray Diffraction (XDR) pattern of the sample: 1 = magnesioferrite, 2 = quartz, 3 = hematite, 4 = zincite, 5 = graphite, 6 = calcium magnesium silicate, 7 = calcite, 8 = wüstite

The sample shows a broad peak at 28° and a weaker one at 43°, suggestive of an amorphous graphitic structure, commonly found in coal and coke-containing materials (Manoj, 2016). This is likely attributed to the feedstock's coal content. The presence of

graphite might arise from coal graphitization, a process catalyzed in the blast furnace at above 1200 °C (Hongtao Li *et al.*, 2020).

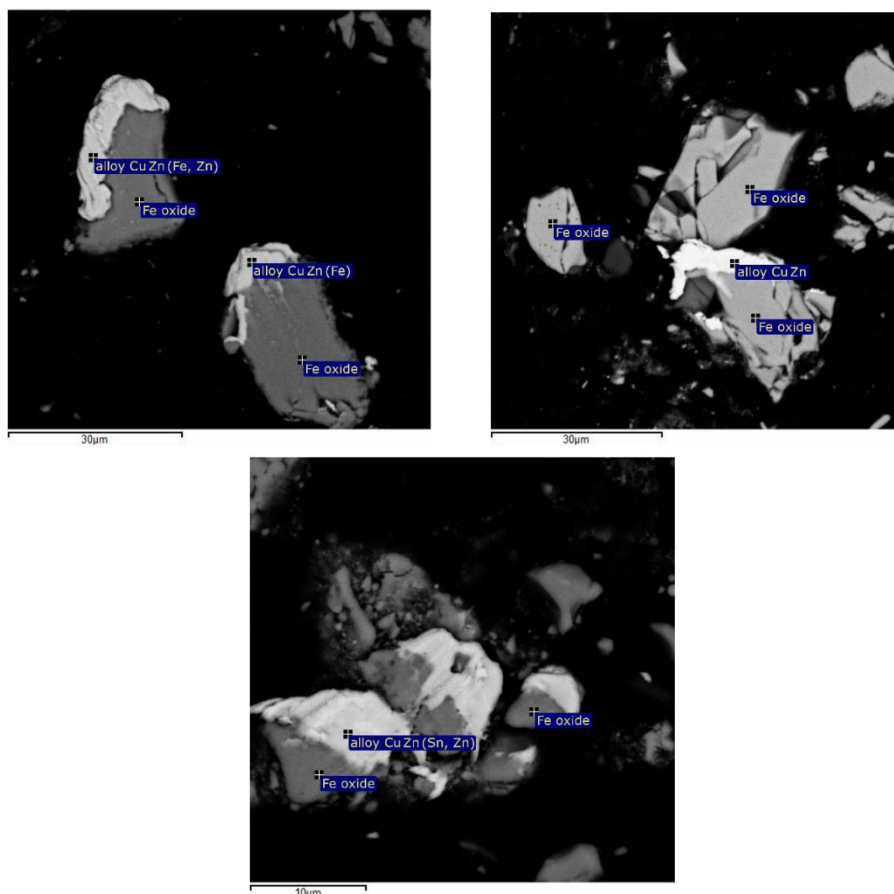
Optical and electron microscopy of the sample revealed that iron-containing phases include hematite, magnetite, magnesioferrite, spinel, silicates, pyrrhotite, and isolated pyrite grains (see Figure 22). The material's angular form hints at formation predominantly through fretting. Iron-containing particles measure up to 0.15 mm, while iron sulphide particles range between 0.020 and 0.010 mm.



**Figure 22: Photomicrograph in reflected light of sludge sample**



The sample also contains copper-zinc alloy particles, occasionally incorporating iron and tin, which merge with iron oxides as depicted in Figure 23. Notably, carbon-containing particles typically lack iron and zinc mineral inclusions, except for neighboring iron sulphides (pyrrhotite and pyrite).



**Figure 23: SEM images of the sludge sample**

Chemical composition analysis of the BFS sample, detailed in Table 5, reveals iron (33.8%) and carbon (34.1%) as primary constituents, with silicon oxide making up 5.05%. Coke and coal particles in the charge, subject to abrasion, likely contribute to the material's sulfur content (Stewart *et al.*, 2022). Furthermore, the sample contains 2.14% zinc, largely in oxidized mineral form. Such elevated zinc content necessitates preliminary treatment before returning the sludge to the blast furnace, considering typical zinc load restrictions of 100 to 200g per ton of liquid metal (Stewart *et al.*, 2022). Zinc predominantly exists in oxide form, suggesting potential for hydrometallurgical sludge processing due to the low presence of zinc ferrite, a mineral that's challenging to extract.

Composition [wt. %]	
Cu	< 0.0010
Ni	0.0074 ± 0.0012
Fe	33.8 ± 0.7
Cr	< 0.02
Mn	0.141 ± 0.030
Mg	0.527 ± 0.010
Al	0.62 ± 0.08
Zn	2.14 ± 0.18
ZnO	1.78 ± 0.21
ZnFe <sub>2</sub> O <sub>4</sub>	< 0.05
Pb	0.055 ± 0.015
Ca	1.64 ± 0.14
SiO <sub>2</sub>	5.05 ± 0.30
S	0.87 ± 0.07
C	34.1 ± 0.4
Cl <sup>-</sup>	0.141
F <sup>-</sup>	0.038 ± 0.015

**Table 5: Chemical analysis of the sludge sample**

Particle size analysis results, outlined in Table 6, reveal that particles smaller than 45 micrometres account for half of the total dust volume and hold over 80% of the zinc, aligning with comparable analysis (Trinkel *et al.*, 2016). Considering zinc's predominance in finer fractions, exploring beneficiation processes seems plausible.

Sieve size [µm]	Mass fraction [%]	Zn content [wt. %]	Zn distribution [%]
100	27.67	0.97	11.75
71	8.60	0.57	2.15
45	12.68	0.85	4.72
Undersize	51.05	3.64	81.38
Total	100	2.28	100

**Table 6: Particle size distribution**

### 3.3 Beneficiation of the sludge

Usage of classification enables an 84% zinc recovery from the 0.045 mm fraction, potentially reducing the recycled material volume by nearly half, as illustrated in Table 7. This minimizes the sludge quantity, with the underflow either being landfilled or returned to the main blast furnace plant, while the overflow is forwarded for additional leaching. Beneficiation is planned to be carried out using a hydrocyclones.

Sieve size [ $\mu\text{m}$ ]	Mass fraction [%]	Composition [wt. %]			
		Zn	Fe	C	Ca
45	48.98	0.67	22.50	18.66	2.10
Undersize	51.02	3.32	44.45	45.20	0.92
Total	100	2.02	33.70	32.20	1.50

**Table 7: Results of wet sieve analysis**

Magnetic separation techniques did not yield anticipated outcomes for this material sample due to insufficient magnetic particle extraction. This could be attributed to a significant portion of the iron in the material being present as hematite, a non-magnetic compound, while the magnetic fraction, comprising magnetite and metallic iron, forms just a minor part.

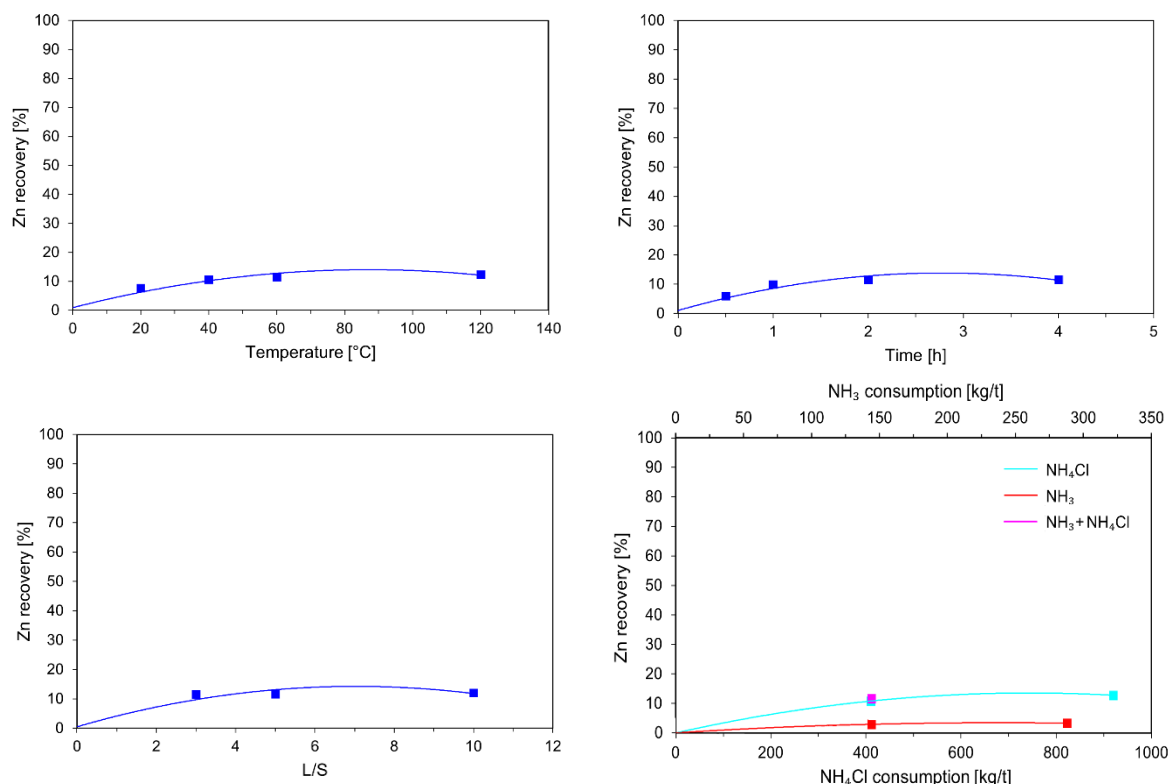
### 3.4 Leaching in ammonia-ammonium chloride

A series of experiments were performed to evaluate the effectiveness of ammonium chloride leaching. The objective of these experiments was to identify the optimum process parameters for achieving maximum zinc extraction while minimizing iron transfer into the solution. This was accomplished by varying a specific process parameter (time, temperature, L/S ratio, and leaching reagent consumption) while keeping the remaining others constant (see Table 8).

Time [h]	Temperature [ $^{\circ}\text{C}$ ]	Consumption [kg/t]		S/L	Yield [%]	Zn content [wt. %]	Zn recovery [%]
		$\text{NH}_3$	$\text{NH}_4\text{Cl}$				
0.5	80	144	410	2	84.35	2.39	6.04
1					84.01	2.06	9.93
2					84.19	1.89	11.47
4					83.98	1.84	11.63
					82.28	2.28	7.75
					82.55	2.03	10.63
					82.69	1.98	11.56
					83.58	1.89	12.35
					89.67	2.33	2.94
					89.48	2.36	3.32
2	80	144	410	2	81.54	1.97	11.53
					83.09	2.04	10.73
					81.60	1.94	12.69
					84.04	1.94	11.56
					83.54	2.12	11.58
					83.80	2.03	12.02

**Table 8: Leaching experiments in ammonia-ammonium chloride**

However, as depicted in Figure 24, zinc recovery rate is only 13%, which is significantly below the desired benchmarks for successful implementation of the technology. These findings suggest that the ammonia-chloride leaching method does not offer optimal conditions for treating this particular type of dust.



**Figure 24: Effect of leaching process parameters on zinc recovery efficiency**

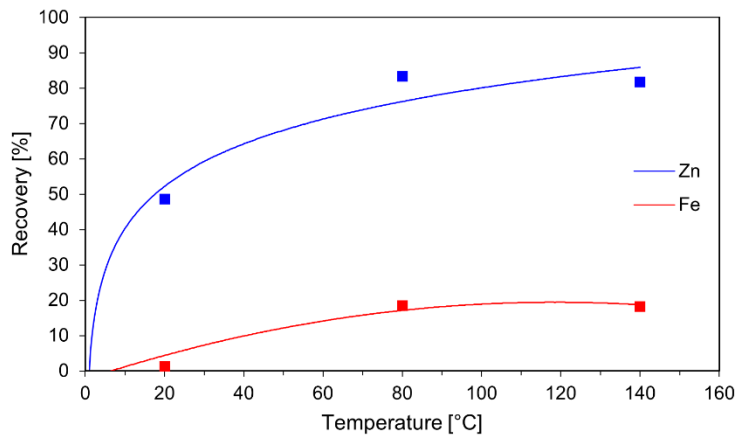
### 3.5 Leaching in sulfuric acid

A similar series of experiments was conducted for sulphuric acid leaching. Based on the literature review and practical technological experience, the parameters for the baseline experiment were determined: acid consumption of 240 kg/t, L/S ratio of 3 and process duration of 2 hours.

Experiments were performed to investigate the effect of temperature variation in the range of 20 to 140 °C with a step of 60 °C (see Table 9). The results showed that zinc recovery into the solution increased up to a plateau as the temperature increased, as shown in Figure 25. Therefore, the optimal temperature of 80 °C was selected.

Temperature [°C]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
20	79.86	4.02	25.58	48.66	1.39
80	75.33	2.79	23.61	83.35	18.58
140	74.27	3.12	24.49	81.74	18.31

**Table 9: Sulphuric acid leaching efficiency under different temperature**

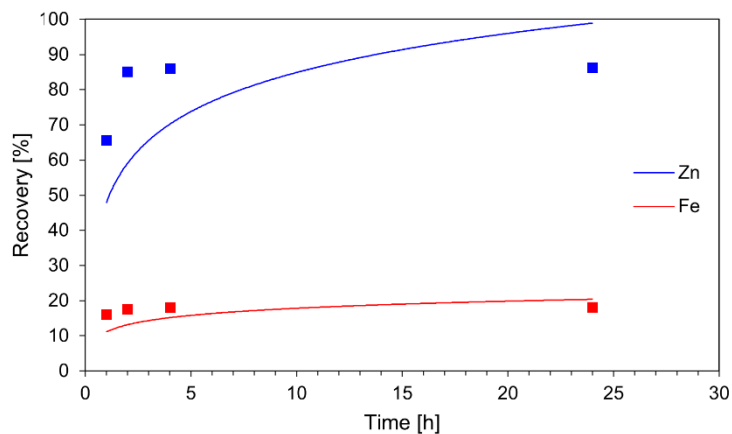


**Figure 25: Effect of temperature on sulfuric acid leaching efficiency**

Further experiments were carried out to assess the impact of leaching duration at the predetermined temperature of 80 °C (see Table 10). The time options tested were 1, 2, 4, and 24 hours. It was observed that increasing the sulphuric acid leaching duration up to 2 hours led to an increase in the extraction rate of zinc in the leaching solution from 65.58% to 85.17%. However, the extraction rates of zinc and iron remained relatively stable as the duration of the process was extended, as shown at the Figure 26. Thus, for further optimization of the process parameters, a leaching duration of 2 hours would be preferable.

Time [h]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
1	81.23	3.02	24.37	65.58	16.09
2	75.28	2.24	24.51	85.17	17.70
4	74.89	2.11	23.92	86.04	18.07
24	74.53	2.10	23.87	86.33	18.14

**Table 10: Sulphuric acid leaching efficiency under different time**

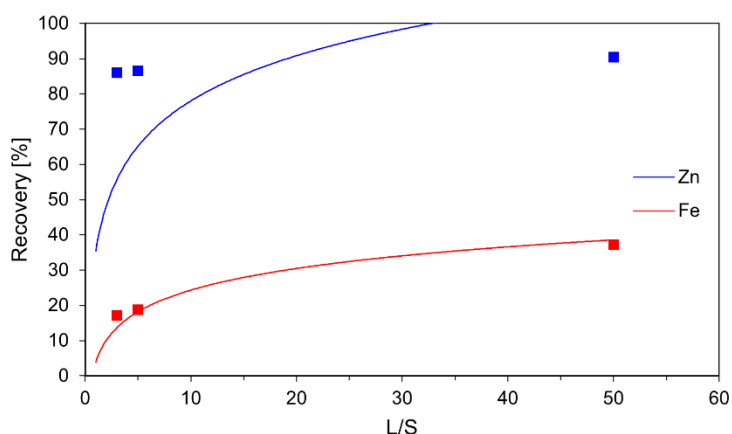


**Figure 26: Effect of duration on sulfuric acid leaching efficiency**

To determine the optimal L/S ratio from the sets 3, 5, and 50, experiments were conducted using the previously selected parameters of 2 hours duration and a temperature of 80 °C (see Table 11). No significant effect on the degree of zinc recovery was observed when varying the L/S ratio, except for experiments with high solution dilution, as shown in Figure 27. However, increasing the dilution of the slurry would require larger apparatus volume, resulting in higher operating costs and increased iron transfer into the solution. Therefore, a L/S ratio of 3 was determined to be the optimal parameter.

L/S	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
3	75.23	2.88	23.43	86.01	17.27
5	75.51	2.75	22.93	86.57	18.84
50	73.63	2.35	20.09	90.51	37.32

**Table 11: Sulphuric acid leaching efficiency under different L/S ratio**



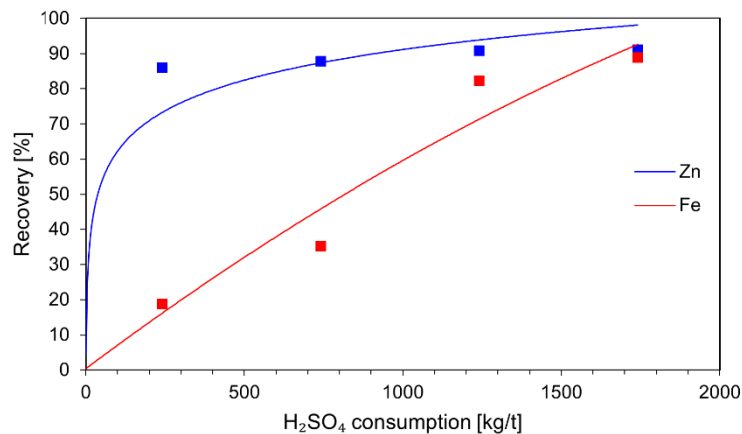
**Figure 27: Effect of dilution on sulfuric acid leaching efficiency**

Using the experimentally verified parameters of 2 hours duration, temperature of 80 °C, and L/S ratio of 3, the acid consumption was varied at 240, 740, 1240, and 1740 kg per 1 tonne of dust (Table 12). The zinc recovery in the sulphuric acid leaching solution reached a peak of 91%, while approximately 80–90% of iron was transferred to the solution, as illustrated in Figure 28.

Additionally, the formation of gypsum ( $\text{CaSO}_4$ ) causes the accumulation of sulphur, which not only hinders acid regeneration but also suppresses the return of iron-containing cake to the blast furnace. This is because sulphur is one of the most detrimental impurities in pig iron, forming a eutectic with iron that has the lowest melting point, making red-shortness. Together with the elevated temperature, all these factors render the process unfavourable.

H <sub>2</sub> SO <sub>4</sub> consumption [kg/t]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
240	74.62	3.36	25.52	86.07	18.82
740	69.97	2.87	23.22	87.84	35.29
1240	68.61	1.83	13.52	90.87	82.26
1740	64.89	1.79	12.82	91.08	88.87

**Table 12: Sulphuric acid leaching efficiency under different H<sub>2</sub>SO<sub>4</sub> consumption**



**Figure 28: Effect of H<sub>2</sub>SO<sub>4</sub> consumption on sulfuric acid leaching efficiency**

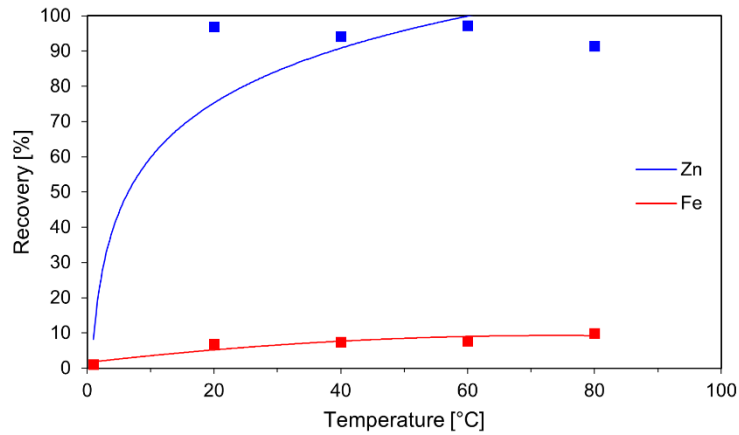
### 3.6 Leaching in hydrochloric acid

Similar experimental procedure was employed for testing hydrochloric acid leaching. The basic parameters were as follows: a duration of 2 hours, L/S ratio of 5, and an acid consumption of 420 kg/t. The temperature ranged from 20 to 80 °C in 20-degree intervals (see Table 13). Increasing the leaching temperature does not yield significant advantages since the zinc extraction efficiency remains nearly unchanged within this temperature range, as depicted in Figure 29.

Investigating higher temperatures, which would require the use of autoclaves, appears impractical due to the heightened aggressiveness of sulfuric acid and the subsequent increase in the cost of the main technological equipment, leading to reduced process profitability. Consequently, a temperature of 20 °C seems to be the most optimal for sulfuric acid leaching of zinc dust.

Temperature [°C]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
20	71.78	0.85	53.02	96.82	6.89
40	71.62	0.86	52.95	94.13	7.45
60	71.23	0.93	52.52	97.20	7.68
80	71.06	0.87	51.89	91.41	9.94

**Table 13: Hydrochloric acid leaching efficiency under different temperature**

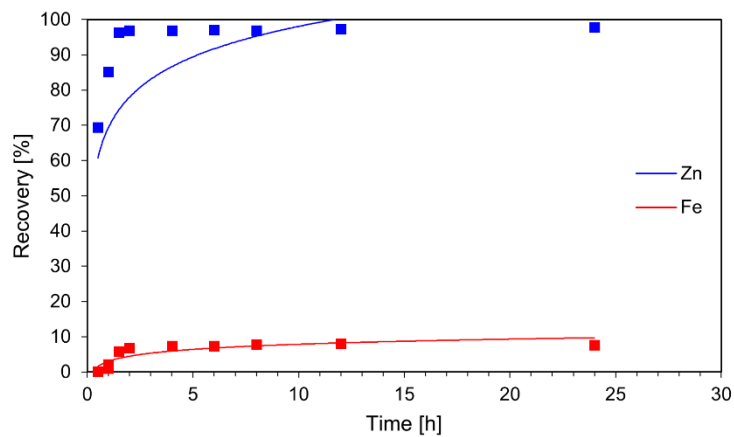


**Figure 29: Effect of temperature on hydrochloric acid leaching efficiency**

To evaluate the influence of process duration on the extraction efficiency of Zn and Fe, a 24-hour experiment was conducted at a temperature of 20 °C with periodic sampling of solution for analysis (see Table 14). The obtained results indicate that the optimal leaching duration is 2 hours, as further prolongation of the process does not contribute to an increase in zinc extraction efficiency as shown in Figure 30.

Time [h]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
0.5	83.69	0.94	46.48	69.42	0.11
1	87.17	0.88	49.76	85.19	2.20
1.5	71.88	0.83	52.70	96.46	5.79
2	71.63	0.88	52.86	96.88	6.91
4	71.54	0.87	52.66	96.90	7.39
6	71.34	0.85	52.66	97.04	7.37
8	71.28	0.89	52.61	96.89	7.74
12	71.25	0.83	52.43	97.34	7.99
24	71.14	0.91	52.58	97.80	7.71

**Table 14: Hydrochloric acid leaching efficiency under different time**



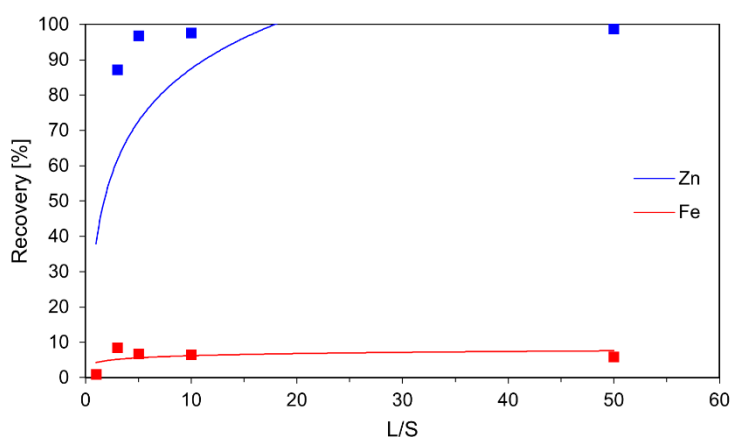
**Figure 30: Effect of duration on hydrochloric acid leaching efficiency**



The determination of the L/S ratio was conducted over a period of two hours, with variations in the pulp dilution level (see Table 15). According to the obtained results, the most favorable L/S ratio for zinc extraction is 5. Reducing this ratio decreases the zinc extraction efficiency from 97% to 87% as illustrated in Figure 31. Conversely, increasing the L/S ratio is not justified as it leads to a significant increase in water consumption in the process, with only a slight improvement in zinc extraction, thereby raising process costs.

L/S	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
3	76.63	1.07	50.33	87.15	8.45
5	74.16	0.95	52.85	96.92	6.85
10	73.84	0.88	53.06	97.70	6.58
50	72.53	0.82	54.10	98.83	5.97

**Table 15: Hydrochloric acid leaching efficiency under different L/S ratio**



**Figure 31: Effect of dilution on hydrochloric acid leaching efficiency**

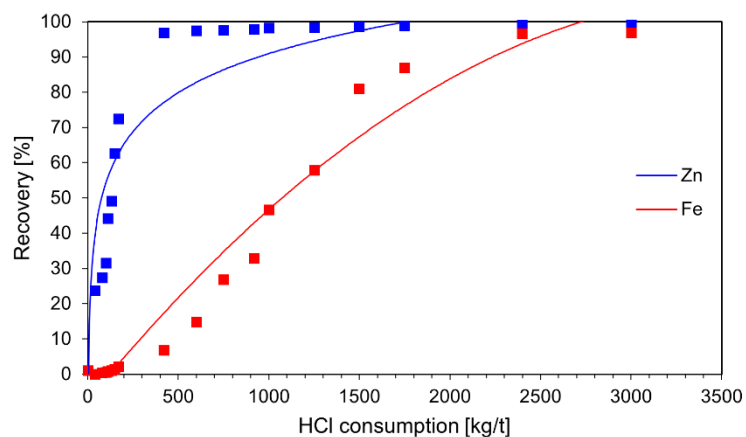
The influence of acid consumption on the leaching process dynamics was studied by varying the HCl consumption in the range from 40 to 2397,5 kg/t (the latter corresponds to the use of concentrated acid at a given L/S ratio). An additional experiment was conducted with an increased acid flow rate of 3000 kg/ton at an L/S ratio of 10 (see Table 16).

From the obtained results, it can be concluded that the sulfuric acid leaching method achieves zinc extraction of up to 99% when using more than 2000 kg/t of acid per ton of sludge. However, in this case, practically all iron, calcium, and other impurities also dissolve into the solution, as shown in Figure 32. The resulting pulp becomes nearly impermeable to filtration. The solid residue mainly consists of silicon oxide and carbon.

HCl consumption [kg/t]	Cake yield [%]	Content in the cake [wt. %]		Extraction [%]	
		Zn	Fe	Zn	Fe
40	88.14	2.80	49.70	23.66	0.00
80	85.16	2.69	50.68	27.40	0.38
100	80.47	2.51	51.27	31.58	0.59
110	79.32	2.10	51.50	44.19	0.78
130	77.41	1.85	51.76	49.10	1.09
150	73.28	1.67	51.92	62.72	1.42
170	71.50	1.20	52.40	72.45	2.10
420	68.73	0.80	52.80	96.76	6.82
600	58.31	0.58	54.33	97.46	14.92
750	55.92	0.62	48.68	97.59	26.88
920	50.68	0.59	47.05	97.93	32.90
1000	44.58	0.40	44.43	98.28	46.64
1250	37.93	0.39	40.25	98.42	57.88
1500	31.64	0.30	21.85	98.64	81.08
1750	24.28	0.27	19.68	98.92	86.90
2398	14.48	0.14	6.76	99.08	96.71
3000	12.02	0.06	8.08	99.12	96.83

**Table 16: Hydrochloric acid leaching efficiency under different HCl consumption**

Considering the aforementioned results, the most favourable choice would be to use an acid quantity of 420 kg/t. With this flow rate, zinc extraction of up to 97% can be achieved, with only around 9% of iron entering the solution. An additional advantage is the possibility of selectively precipitating iron through ammonia neutralization. Moreover, this acid consumption allows for a relatively high level of acid regeneration.



**Figure 32: Effect of HCl consumption on hydrochloric acid leaching efficiency**

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## 4 Development of hydrometallurgical technology for the processing of blast furnace sludges from PAO Severstal

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### 4.1 Leaching process

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Based on the research findings and economic considerations, the optimal amount of hydrochloric acid was determined to be 420 kg/t. Based on the leaching data of the investigated materials and aiming to approach industrial conditions, a counter current two-stage leaching scheme is proposed: initial leaching in the recirculating solution obtained after the second leaching stage, followed by subsequent leaching with the addition of fresh acid. The technological parameters for each stage are provided in Table 17. This technology ensures the most efficient separation of iron and zinc, as well as the maximum zinc extraction into the solution with an optimal hydrochloric acid consumption.

Parameter	1 <sup>st</sup> stage	2 <sup>nd</sup> stage
Reagent	solution from the second leaching stage	HCl
Temperature	20 °C	
Time	2 h	
L/S	5	
Reagent consumption	440 kg/t	

**Table 17: The technological parameters of the leaching process**

In laboratory conditions, an experiment was conducted to determine the efficiency and characteristics of the proposed leaching scheme. The recirculating solution for the first leaching stage was prepared separately under conditions similar to the parameters of the second leaching stage. After each leaching stage, the pulp was subjected to filtration, and the solution and residue were analysed for Zn, Fe, and Ca content. The loading for the second leaching stage was taken from the residue obtained after the first leaching stage.

Table 18 presents the data on the leaching of BFS at each leaching stage. This technology allows for the extraction of up to 95% of zinc, indicating high process efficiency. However, it is worth noting that only 31% of the total dust volume generated at CSM is processed. It is important to highlight that there is long-term potential for incorporating the processing of furnace dust into the system. Such an approach would expand the scope of recycling, thereby increasing waste utilization efficiency.

Material/product	Composition [wt. %]			Yield [%]	End-to-end recovery rate [%]		
	Zn	Fe	Ca		Zn	Fe	Ca
Feed material	3.32	44.45	0.92	100			
Dried cake from 1 <sup>st</sup> stage	0.83	42.1	0.66	94	95.36	23.03	75.83
Dried cake from 2 <sup>nd</sup> stage	0.18	40	0.26	85.5			

**Table 18: Efficiency of at each leaching stage**

## 4.2 Selective recovery of elements from the solution

During leaching, high-concentration solutions are produced, containing zinc (Zn) at 6.33 g/L, iron (Fe) at 20.47 g/L, and calcium (Ca) at 1.40 g/L. To selectively separate iron, the method of hydrolytic purification of the solutions was chosen. Since the pH values for iron and zinc hydroxide formation are similar, ammonia was used as a neutralizing reagent to completely precipitate iron. In ammonia-containing solutions, zinc forms an ammonia complex and does not precipitate.

After decontamination of the leaching solution from iron, zinc is extracted in the form of zinc hydroxide by evaporating ammonia from the solution. The addition of calcium oxide or hydroxide to the solution at a ratio of 1 mole of Ca per 1 mole of  $\text{Cl}^-$  ion accelerates the process and ensures maximum zinc recovery.

In the subsequent stage, calcium is removed from the solution while simultaneously regenerating a portion of the acid. This process is achieved through intensive bubbling of the solution with carbon dioxide gas (in industrial-scale production, furnace gases can be utilized) for one hour, leading to the formation of sparingly soluble calcium carbonate.

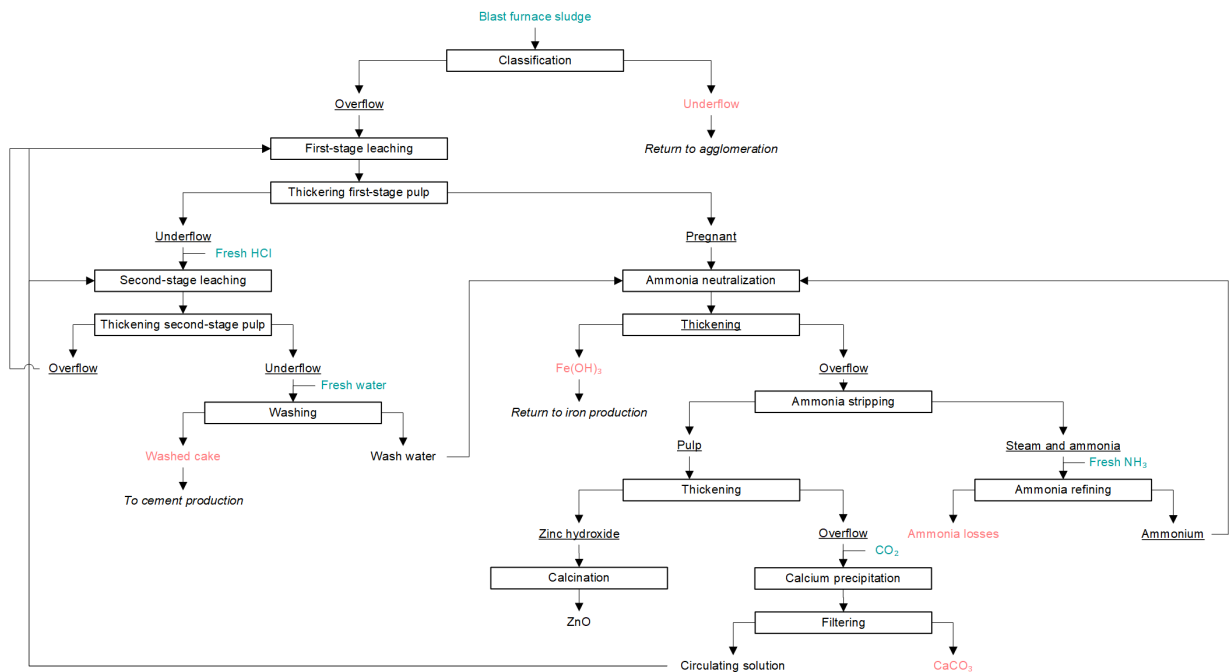
The composition of the solutions obtained at all the aforementioned stages of the technology is presented in Table 19. The decrease in zinc and calcium concentrations during the iron precipitation stage is due to the dilution of the pulp with ammonia. The increase in calcium content during the zinc precipitation stage is associated with the dissolution of the added lime.

Stage	Concentration [g/L]		
	Zn	Fe	Ca
After iron precipitation	3.94	0.53	0.88
After zinc precipitation	0.43	0.57	36.57
After calcium precipitation	0.43	0.57	0.04

**Table 19: Composition of solutions in consecutive stages of selective precipitation**

The solutions obtained after calcium precipitation can be partially recycled, thereby reducing acid consumption. To assess the prospects of complete circulation and accumulation of impurities in the solutions, further research is required on an enlarged scale. Additionally, additional studies on precipitation using real production gases or gases with a similar composition and temperature are necessary to verify the possibility of calcium precipitation with furnace gases.

The final flowsheet proposed for the processing of BFSs is presented in Figure 33.



**Figure 33: Flowsheet of blast furnace sludge processing**

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## 5 Summary and outlook

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The aim of this thesis was to develop a technology for recycling sludge from Severstal's blast furnace operations. Blast furnace sludge, a waste product of the iron and steel industry, contains elevated levels of zinc, making it unsuitable for recycling back into the main production process. The research explored various methods of sludge processing, focusing on both pyro and hydrometallurgical processes.

Considering the environmental impact and sustainability aspects, hydrometallurgical technology emerged as the more favourable option. This technology offers reduced environmental impact, a small carbon footprint, easy recovery of by-products, and the ability to regenerate components. To determine the optimal method, the mineralogical and chemical composition of the sludge was thoroughly analysed. It was found that the sludge contained a low amount of poorly soluble zinc ferrite and mainly zinc in oxide form, which influenced the selection of the leaching reagents.

Through practical experience and a comprehensive literature review, the most promising leaching reagents were identified. Leaching experiments were conducted to determine the optimum parameters for each process. Hydrochloric acid leaching emerged as the most successful method, achieving a remarkable 90% recovery of zinc. Based on this finding, a BFT processing technology was developed specifically for hydrochloric acid leaching.

By successfully developing this recycling technology, the thesis contributes to the sustainable management of blast furnace sludge. It enables the efficient recovery of zinc, a valuable resource, while reducing waste and environmental impact. The development of a closed-loop circuit not only allows for the recovery of valuable zinc resources but also emphasizes the recovery of reagents, creating a more sustainable and efficient recycling system.

In conclusion, the research presented in this thesis provides a valuable contribution to the field of blast furnace sludge recycling. The developed technology for hydrochloric acid leaching offers a practical and sustainable solution for zinc recovery from sludge, allowing for the effective utilization of this waste product. The findings of this study are promoting a more environmentally friendly and economically viable approach to waste management and resource recovery.

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## 9 List of Abbreviations

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BFS	Blast Furnace Sludge
CSM	Cherepovets Steel Mill
CZP	Chelyabinsk Zinc Plant
L/S ratio	Liquid-to-Solid ratio
LME	London Metal Exchange
MHF	Multi-Hearth Furnace
RHF	Rotary Hearth Furnaces
SHG	Special High Grade
XDR	X-Ray Diffraction