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Comparative Study of Steel Mill Dust Leaching with Phosphoric Acid and Sodium Hydroxide

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Abstract: Steel mill dust (SMD), produced by electric arc furnaces, is a highly polluting industrial waste due to its high content of metals (Zn, Fe, and Pb) and fine particle size (ca. 5.4 µm). This residue can be valorized to recover Zn using pyro and hydrometallurgical methods, with hydrometallurgy offering greater selectivity and lower energy costs. However, composition of SMD presents a challenge in identifying an optimal leaching agent. This study investigates the preferential extraction of Zn using two leaching agents, namely 150 g L^{-1} (1.5 M) phosphoric acid (H_3PO_4) and 240 g L^{-1} (6 M) sodium hydroxide (NaOH), in a two-stage leaching process (80 °C). Metallic Zn from the alkaline pregnant solution was recovered by electrodeposition (750 A/m^2, graphite anode, stainless-steel cathode) and smelting (450 °C). The samples of SMD contained 26.3% Zn, 20.1% Fe, and 0.9% Pb, in compounds such as magnetite (Fe_3O_4), zincite (ZnO), and franklinite ($ZnFe_2O_4$). Each leaching agent successfully attained a 99% Zn recovery, demonstrating the proposed procedure's high efficacy. However, H_3PO_4 leached also Fe and corroded the cathode during electrodeposition, thereby restricting the final recovery of metallic Zn. NaOH demonstrated greater selectivity for Zn over Fe and Pb, producing high-purity Zn deposits on the cathode by electrodeposition and 99% metallic zinc by smelting.

Keywords: steel mill dust; acid leaching; alkaline leaching; zinc; phosphoric acid

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1. Introduction

Steel mill dust (SMD) generated from scrap steel smelting is considered one of the most polluting solid wastes in the industry. Dutra et al., Halli et al., and Holappa et al. [1–3] estimated that approximately 12 million tons of SMD are produced each year. However, more than half of this material is sent to landfills, resulting in an annual loss of over a million tons of zinc [4].

During the smelting process, the zinc present in the scrap steel evaporates, oxidizes, and transfers to devices that retain the SMD as solid ZnO. On average, a ton of scrap produces between 15 and 25 kg of SMD, with zinc content varying between 15% and 40%, as reported by Guézennec et al. and Palimakaą et al. [5,6]. According to Ostrowska and Mierzwa [7], SMD also contains other elements such as cadmium, nickel, chromium, manganese, carbon, tin, antimony, and copper. If these residues are not properly managed, they could easily become a source of water and soil pollution. Therefore, it is necessary to propose alternatives to valorize them and use them as raw materials for other processes [2].

The current technology for processing SMD primarily focuses on pyrometallurgical and hydrometallurgical methods. Pyrometallurgical methods use high temperatures to reduce the ZnO content and oxidize the zinc in the gas phase. The dominant method for processing SMD is the Waelz kiln process, which represents 85% of the zinc recovery market [8]. This technology involves the use of carbon monoxide and other reducing agents such as coal, coke, and other carbon sources [9]. However, the Waelz method produces a significant amount of waste that requires further treatment for final disposal [10,11].

Hydrometallurgical treatment of SMD offers advantages over pyrometallurgical methods, such as the potential for smaller-scale operations, lower transportation costs, and lower investment as well as high selectivity for recovering metals of interest [12–14]. Hydrometallurgical methods can be easily scalable and offer the possibility to process metals such as zinc, iron, lead, and cadmium [15].

Traditional leaching agents used in the recovery of zinc from SMD include acids (e.g., sulfuric acid) and alkalis (e.g., sodium hydroxide). In particular, the use of sulfuric acid has attracted attention in the last decade, as high zinc recoveries (>90%) have been achieved by using high temperatures (60–90 °C), different pressures (1–3 atm), or variations in the acid concentrations employed (120–200 g L^{-1}). Additionally, this process has a lower operating cost compared to other alkaline processes that use concentrations of 6 M and 12 M [16–19]. Due to the high iron content in SMD, it is not always possible to recover zinc using traditional hydrometallurgical methods [6]. However, hydrometallurgy is still a promising alternative for zinc recycling due to the control of iron dissolution during the process by selective leaching [13].

SMD generally contains franklinite ($ZnFe_2O_4$), zincite (ZnO), and magnetite (Fe_3O_4). Zincite and magnetite do not present problems in acid or alkaline leaching, but zinc ferrite is considerably refractory to leaching. In fact, zinc recovery from SMD by leaching largely depends on the content of zinc ferrite and the particle size. Due to the complexity of the chemical composition of these residues, it has been challenging to define the best conditions to recover zinc by leaching [20,21].

Leaching of SMD with sulfuric acid is not very common due to the low zinc recoveries obtained. Thus, it is necessary to develop selective and sustainable methods to recycle SMD, taking into account the sample characteristics, especially the high contents of zinc ferrite ($ZnFe_2O_4$, franklinite) [21,22]. Although alkaline leaching seems advantageous due to the low iron leaching, it requires high concentrations of the alkaline leaching medium [23].

While acid leaching does not require high concentrations in the solution, as alkaline leaching does, iron is partially transferred to the solution in the first case. After acid leaching, the remaining zinc is in the form of zinc ferrite [24,25], complicating its subsequent extraction. Acid leaching with sulfuric acid, depending on the conditions used, dissolves the most reactive zinc species, while most of the iron remains in the residue due to the presence of zinc ferrites [13].

In recent years, the thermal pretreatment of SMD has modified the process. This has led to the hydrometallurgical processing of zinc oxide, known as ENDUTEC/EZINEX [26]. Recupac, a Chilean company, developed a recycling patent to extract commercial iron and zinc compounds from electric arc furnace SMD using proven hydrometallurgical technology. Iron and zinc are recovered, and the iron oxide is used to manufacture industrial pigments. Although many processes have been developed in laboratories and on a pilot or larger scale, they have not been implemented in the industry for various reasons, which has delayed their implementation for several years [27].

To date, most studies on the leaching of SMD have used sulfuric acid and sodium hydroxide as leaching agents. Sulfuric acid has several advantages, such as high dissolution speed, availability, and moderate prices. Furthermore, it is possible to use low concentrations and subsequently combine it with the electrolysis process to obtain metallic zinc, with the regeneration of the acid used. However, the main disadvantage of using sulfuric acid as a leaching agent is the lack of selectivity towards other metals present in the SMD, especially iron, which is sometimes present in larger quantities than zinc. This ultimately results in high consumption of the leaching agents needed to purify the solution of large amounts of iron [27].

Phosphoric acid in zinc recovery from SMD has been used in previous studies such as that of Halli et al. [15], with low zinc recoveries of less than 10% despite leaching for 168 h with concentrations of 1.92 M and 19.2 M. Additionally, industrial-grade di-(2-ethylhexyl) phosphoric acid (D2EHPA), 97% pure, was used as an organic solvent for the separation of iron and zinc from SMD [28].

An alkaline leaching agent can be selective towards zinc over iron. Moreover, this selectivity allows the separation of lead from zinc, which can be used as an additional intermediate element and raw material to recover from SMD. However, a disadvantage of this process is that high concentrations of the leaching agent are required to achieve efficient and significant zinc recovery, making it more costly than sulfuric acid and complicating the recovery of the solution [6].

Palimakaą et al. [6], employing SMD with an alkaline leaching agent such as sodium hydroxide, demonstrated a significant dependence on certain leaching parameters, such as temperature and sodium hydroxide concentration, which must be controlled to enhance the efficiency of the process. However, other parameters like the percentage of solids, particle size, and stirring speed were found relatively insignificant for the efficient extraction of zinc in the process.

Ammonium chloride and ammonium carbonate have also been widely used to selectively dissolve zinc from secondary residues by forming ammonium–zinc complexes, leaving iron in the residue. Several processes such as CENIM-LNETI and EZINEX have been developed to utilize ammonium chloride as a leaching agent for zinc recovery. The CENIM process was developed in Spain and Portugal for the treatment of sulfide concentrates and other secondary raw materials. In this process, electric arc furnace dust is leached with ammonium chloride after water washing to solubilize zinc oxide [29].

Zinc is present in SMD in the form of ZnO and $ZnFe_2O_4$; therefore, the key challenge is to extract zinc from both zinc species. Zinc oxide is easily leached, but zinc ferrite ($ZnFe_2O_4$) is a very stable phase, which may result in low zinc recovery [30].

With this background, this study aimed to determine whether either acid leaching (with phosphoric acid) or alkaline leaching (with sodium hydroxide) is selective to recover zinc from SMD. Moreover, the best conditions to recover zinc from pregnant solutions obtained in the leaching experiments were studied by a cementation process followed by subsequent electrodeposition to obtain metallic zinc as the final product.

2. Materials and Methods

For the evaluation of zinc recovery from SMD, a sample of approximately 100 kg of material was taken from the output of the electric furnace of a steel production company located in the Cotopaxi province in central Ecuador. The following methodology was employed.

2.1. Physical, Chemical, and Mineralogical Characterization of SMD

The SMD sample was dried at ambient temperature of 20 °C for 24 h, homogenized, and quartered into 1 kg fractions for subsequent analysis and testing. Physical characterization included the determination of apparent and real density according to standard (ISO8130-3) [31] and particle size measurement using the HORIBA LA-950 V2 (Horiba, Seoul, Republic of Korea) with a refractive index for SMD of 2.234.

Chemical characterization of the SMD was determined by X-ray fluorescence using the Bruker S8 Tiger AX15 instrument (Bruker, Karlsruhe, Germany). This characterization was carried out using pulverized samples (<38 microns) that were pressed into pellets. A sequential wavelength dispersive X-ray fluorescence spectrometer, S8 TIGER 3KW, was used, featuring a high-performance X-ray tube with a front window and a rhodium anode, covering an elemental range from beryllium (Be) to uranium (U). The SPECTRAplus V2 software was used for calibration and evaluation, employing the QuantExpress method.

Mineralogical characterization was performed using X-ray diffraction using the Bruker AXS D8 Advance model (Bruker, Karlsruhe, Germany). This characterization was performed using pulverized samples (<38 microns) on a D8 ADVANCE X-ray diffractometer, with a CuKα wavelength of 1.5418 Å, equipped with a graphite monochromator. Measurements were taken from 3° to 70° (2θ) with a step size of 0.02° and a scan speed of 1 min per degree. Qualitative and semi-quantitative phase identification was conducted using the Diffractplus software (Eva and Topas 2.0). The different mineralogical phases were identified by comparison with X-ray diffraction patterns from the International Center for Diffraction Data (ICDD) database.

2.2. Evaluation of Zinc Recovery from Steel Mill Dust by Acid and Alkaline Leaching

Zinc recovery was evaluated using two leaching agents: phosphoric acid and sodium hydroxide. Additionally, leaching tests with sulfuric acid were also conducted to obtain reference values with this leaching agent that was previously employed for zinc recovery in traditional hydrometallurgical methods.

2.2.1. Acid Leaching in Two Stages

The leaching variables were selected according to the study presented by Ricaurte et al. [32]. The leaching process was carried out in two stages. Initially, 40 g of SMD were leached at 10% solids with heating to a temperature of 80 °C, using analytical-grade phosphoric acid 85 wt.% purity from Sigma Aldrich Manufactured in St. Louis, Missouri, United States. at a concentration of 150 g L^{-1} (1.5 M), with magnetic stirring for 5 h. After the first stage, the solution was filtered, resulting in the separation of the residue, the pregnant solution, and the washing solution. The residue was then dried (60 °C for 8 h), weighed, and subjected to a second stage of leaching under the same conditions. Following the 5 h leaching process in the second stage, the solution was filtered again to separate the pregnant solution and the washing solution. The residue was dried and weighed.

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The pregnant and washing solutions were sent for analysis of Zn, Fe, and Pb by atomic absorption spectrometry on a Perkin Elmer AA 300 instrument (Perkin Elmer, Shelton, CT, USA), while the residue was sent for analysis by X-ray fluorescence on the Bruker S8 Tiger AX15 instrument (Bruker, Karlsruhe, Germany).

The same procedure was followed using analytical-grade sulfuric acid 98% purity from Fisher Scientific at a concentration of 150 g L^{-1} (1.5 M) as leaching agent.

2.2.2. Alkaline Leaching with Sodium Hydroxide in Two Stages

The leaching variables were selected according to the study presented by Widi et al. and Palimakaą et al. [4,6]. The leaching process was carried out in two stages. Initially, 40 g of SMD were leached at 10% solids, heated to a temperature of 80 °C, using analytical-grade sodium hydroxide 98% purity from Fermont at a concentration of 240 g L^{-1} (6 M), with magnetic stirring for 5 h. After the first stage, the solution was filtered, separating the residue, the pregnant solution, and the washing solution. The residue was then dried, weighed, and subjected to a second stage of leaching under the same conditions. Following the 5 h leaching process in the second stage, the solution was filtered again to separate the pregnant solution and the washing solution. The residue was dried (60 °C for 8 h) and weighed. The same procedure was repeated with the same leaching conditions, varying the solid percentage to 20%, 30%, and 40%, respectively.

At the end of the leaching tests, the zinc, iron, and lead content in the pregnant and the washing solutions was determined by atomic absorption on a Perkin Elmer AA 300 instrument (Perkin Elmer, Shelton, CT, USA). The remaining zinc in the solid sample was determined by X-ray fluorescence on the Bruker S8 Tiger AX15 instrument (Bruker, Karlsruhe, Germany).

2.3. Evaluation of Zinc Recovery by Electrodeposition from Leaching Solutions

The pregnant solutions obtained from the leaching experiments with phosphoric acid and sodium hydroxide were subjected to zinc recovery by electrodeposition. Before this process, a cementation with zinc powder was performed, aimed at reducing the concentration of metals such as iron and lead, which were metals also present in the leaching solution. The cementation was conducted adding 5 g of metallic zinc powder at 60 °C on a heating plate with magnetic stirring for 2 h.

The electrodeposition process was carried out in cells with a volume of 516 cm^3, and the cell dimensions were 140 mm × 82 mm × 45 mm. The anode/cathode separation was precisely set to 25 mm to optimize the deposition of zinc on the cathode. The electrolyte used in the electrodeposition process was the pregnant solution obtained from the leaching and treated by cementation with zinc powder, which primarily consisted of sodium hydroxide. Importantly, no organic compounds were present in the electrolyte, ensuring the selectivity and purity of the zinc deposited.

The variables used for electrodeposition were adapted with some modifications from the work done by Palimakaą et al. and Ricaurte et al. [6,32]. The experiment was conducted with current intensities of 750 A m^{-2} and 1000 A m^{-2}. pH control was implemented to prevent the dilution of zinc at the cathode, with pH levels maintained between 0 and 2 for pregnant solutions of phosphoric acid and between 9 and 11 for pregnant solutions of sodium hydroxide. The electrodeposition time ranged from 0.5 to 2 h until metallic zinc was deposited on the anode. Three different materials were used as anodes: lead, graphite, and stainless steel. The cathodes in all the experiment were made of stainless steel.

The remaining zinc, iron, and lead content in the electrolyte after the electrodeposition was determined by atomic absorption on a Perkin Elmer AA 300 instrument (Perkin Elmer, Shelton, CT, USA).

The chemical composition of the metallic zinc deposited on the cathode was dried, washed, and analyzed by X-ray diffraction using the Bruker AXS D8 Advance model (Bruker, Karlsruhe, Germany) and by X-ray fluorescence using the Bruker S8 Tiger AX15 instrument (Bruker, Karlsruhe, Germany). A final stage of zinc purification involved smelting with ammonium chloride 99.5% purity from Sigma Aldrich and analytical-grade potassium chloride 99% purity from Sigma Aldrich. This process is crucial for refining the zinc and eliminating any remaining impurities. Ammonium chloride and potassium chloride were used as fluxing agents at 75% by weight relative to the sample weight. The smelting process was conducted at 450 °C for 2 h.

The summary of the steel mill dust treatment methodology is presented in the diagram in Figure 1.

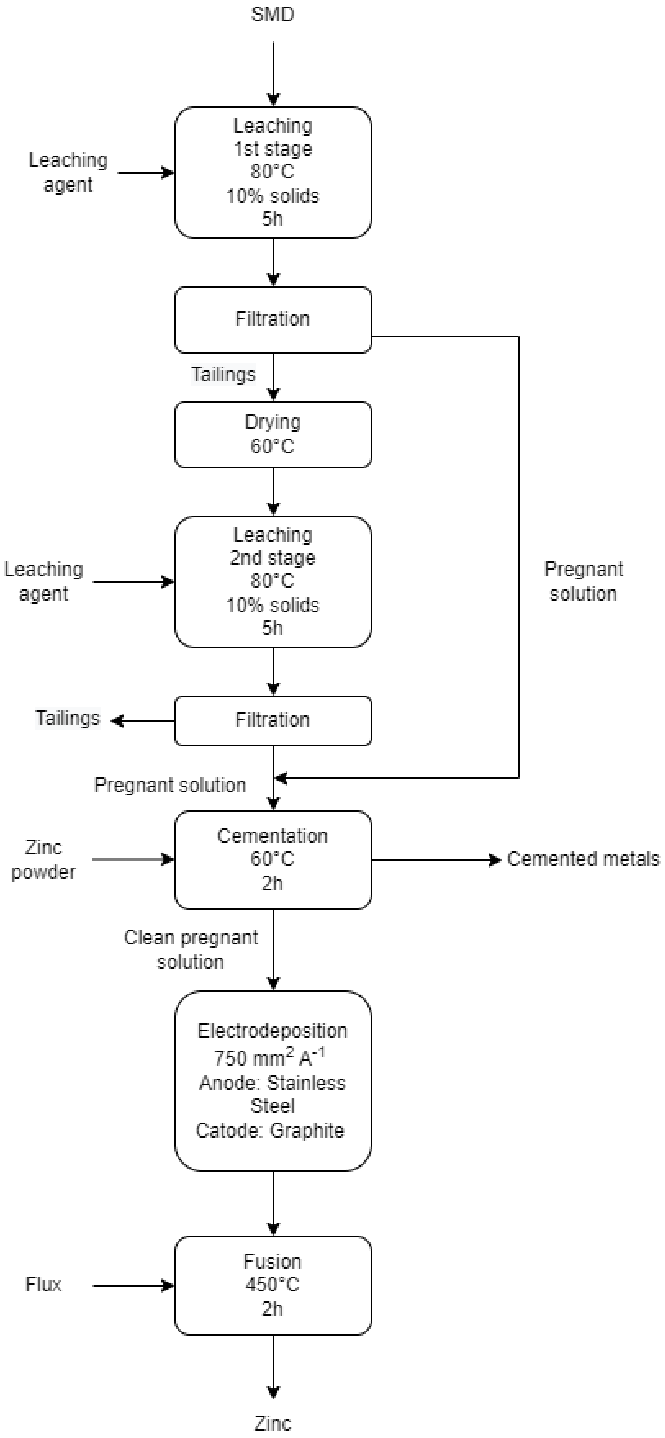

Figure 1. Flowchart of the comparative study of steel mill dust using phosphoric acid and sodium hydroxide as leaching agents.

3. Results

3.1. *Physical, Chemical, and Mineralogical Characterization of Steel Mill Dust*

Regarding the physical characterization, values of 0.8 g cm^{-3} for apparent density and 3.22 g·cm^{-3} for real density were obtained. Concerning the particle size, it was found that the d_{80} was 5.4 µm, while the d_{50} was 2.5 µm. The particle size distribution curve is presented in the Figure 2.

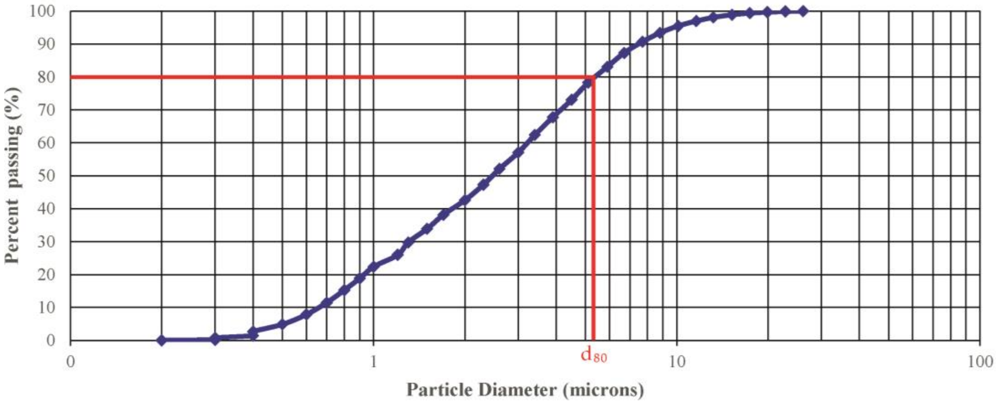

Figure 2. Particle size distribution curve of the SMD.

In terms of chemical characterization by XRF, it was identified that the SMD mainly contains zinc (26.3%) and iron (20%), followed by smaller amounts of calcium (3.7%), magnesium (3.4%), sodium (3.1%), and lead (0.9%), as shown in Table 1.

Table 1. Chemical Characterization Results by XRF of SMD.

Element	Content (%)
Zn	26.3
Fe	20.0
Ca	3.7
Mg	3.4
Na	3.1
Pb	0.9
Al	0.3

As presented in Table 2, mineralogical characterization by XRD revealed the presence of magnetite (42%), zincite (38%), and franklinite (4%) as the most representative compounds in the SMD. Moreover, 8% sodium chloride (halite) was another component present in the SMD, and the XRD spectrum is shown in Figure 3.

Table 2. XRD Analysis Results of SMD.

Mineral	Formula	Content (%)
Magnetite	Fe_3O_4	42
Zincite	ZnO	38
Halite	NaCl	8
Franklinite	$ZnFe_2O_4$	4

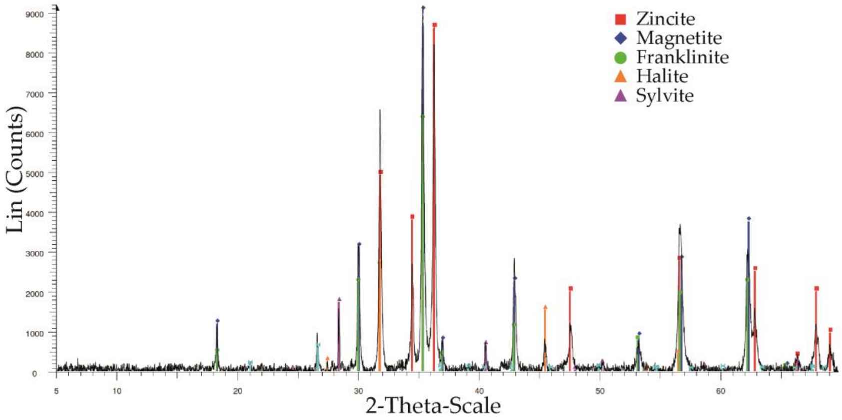

Figure 3. X-ray diffraction analysis of the SMD (red: zincite, ZnO; blue: magnetite, Fe_3O_4; green: franklinite, $ZnFe_2O_4$; orange: halite, NaCl; purple: sylvite, KCl).

3.2. Results of the Evaluation of Zinc Recovery from Steel Mill Dust by Leaching

3.2.1. Acid Leaching Tests

The efficiency of leaching SMD using sulfuric acid and phosphoric acid was evaluated employing the methodology described in Section 2.2.1.

For two-stage leaching with sulfuric acid at 80 °C, 10% solids, pH = 2, and 5 h of agitation per stage, zinc recovery was 97% in the first stage and 2.9% in the second stage, totaling 99.9% zinc recovery. Lead recovery was 0.5% in two stages of leaching, and iron recovery was 98.7% in two stages, as presented in Table 3. The stoichiometric reaction of zincite with sulfuric acid is shown in Equation (1) as presented by Ricaurte et al. [32], while the reaction of sulfuric acid with franklinite is shown in Equation (2) as presented by Havlík et al. [24].

$$ZnO + H_2SO_4 \rightarrow ZnSO_4 + H_2O \tag{1}$$

$$ZnFe_2O_4 + 4H_2SO_4 \rightarrow ZnSO_4 + 2FeSO_4 + 4H_2O \tag{2}$$

Table 3. Zinc, lead, and iron recovery from SMD with sulfuric acid after two stages of leaching at 80 °C, 150 g L^{-1} (1.5 M) sulfuric acid, 10% solids, and 5 h of agitation per stage. The first stage is with sulfuric acid, and the second stage is with sulfuric acid.

Metal Recovery (%)	H_2SO_4	
	1st Stage	**2nd Stage**
Zn	97	2.9
Pb	0.4	0.1
Fe	74.0	24.7

Under the same leaching conditions using phosphoric acid, zinc recovery was 97% in the first stage and 3.1% in the second stage, totaling 100% recovery. Lead recovery was 1.2% in two stages of leaching, and iron recovery was 7.2% in two stages, as presented in Table 4. The stoichiometric reaction of zincite with phosphoric acid is presented in Equation (3), while the reaction of phosphoric acid with franklinite is shown in Equation (4). These two equations are presented as a hypothesis of the possible leaching behavior of phosphoric acid taking into consideration the work of Kuzin et al. [33].

$$ZnO + 2H_3PO_4 \rightarrow Zn(H_2PO_4)_2 + H_2O \tag{3}$$

$$ZnFe_2O_4 + 4H_3PO_4 \rightarrow Zn(H_2PO_4)_2 + 2FePO_4 + 2H_2O \tag{4}$$

Table 4. Zinc, lead, and iron recovery from SMD in two stages with phosphoric acid 150 g L^{-1} (1.5 M), heating at 80 °C, 10% solids, and 5 h of agitation in both stages. The first stage is with phosphoric acid, and the second stage is with phosphoric acid.

Metal Recovery (%)	H_3PO_4	
	1st Stage	**2nd Stage**
Zn	96.5	3.1
Pb	0.6	0.6
Fe	3.7	3.5

3.2.2. Alkaline Leaching Tests

Zinc recovery with alkaline leaching at 80 °C with sodium hydroxide (240 g L^{-1}—6 M) is presented in Figure 4. The highest value of zinc recovery was 80% for 10% solids, and pH = 11. Higher solid percentages did not increase the zinc recovery values under the same leaching conditions. In fact, values of 74%, 58%, and 34% were obtained for leaching at 20%, 30%, and 40% solids, respectively. Additionally, this lower solids concentration helped to reduce viscosity and improve the flow of the solution, leading to better mixing and more uniform leaching conditions. In the following leaching tests, a value of 10% of solids was selected due to these higher recoveries obtained. The stoichiometric reaction of zincite with sodium hydroxide is presented in Equation (5), while the reaction of sodium hydroxide with franklinite is shown in Equation (6). Both equations are presented in the study of Palimakaą et al. [6].

$$ZnO + 2NaOH \rightarrow Na_2ZnO_2 + H_2O \quad (5)$$

$$ZnFe_2O_4 + 8NaOH \rightarrow Na_2ZnO_2 + 2NaFeO_2 + 4H_2O \quad (6)$$

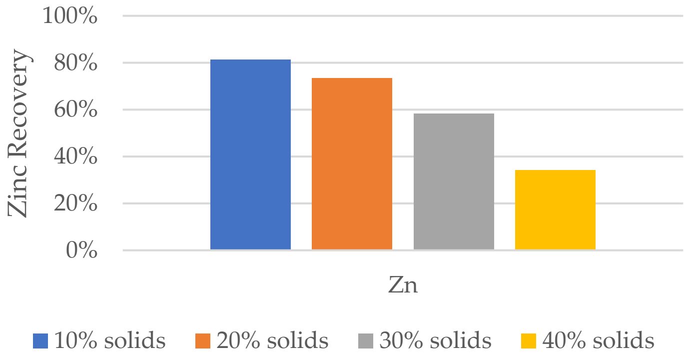

Figure 4. Zinc recovery from SMD with sodium hydroxide at 10%, 20%, 30%, and 40% solids and 5 h of agitation, heating at 80 °C, and a sodium hydroxide concentration of 240 g L^{-1} (6 M).

Using two stages, 10% solids, pH = 11, and heating at 80 °C, zinc recovery was 79.2% in the first stage and 20% in the second stage, totaling 99.2% zinc recovery. Lead recovery was 79.5% in two stages of leaching, and iron recovery was 0.02% in two stages, as presented in Table 5.

Table 5. Zinc, lead, and iron recovery from SMD with sodium hydroxide (240 g L^{-1}—6 M) in two stages: heating at 80 °C, 10% solids, and 5 h of agitation in both stages. The first stage is with sodium hydroxide, and the second stage is with sodium hydroxide.

Metal Recovery (%)	NaOH	
	1st Stage	2nd Stage
Zn	79.2	20
Pb	60.6	18.9
Fe	0.01	0.01

A summary of the values of recovery percentage of Zn, Pb, and Fe obtained by leaching with H_3PO_4 (150 g L^{-1}—1.5 M), NaOH (240 g L^{-1}—6 M), and H_2SO_4 (150 g L^{-1}—1.5 M) in two stages is presented in Figure 5.

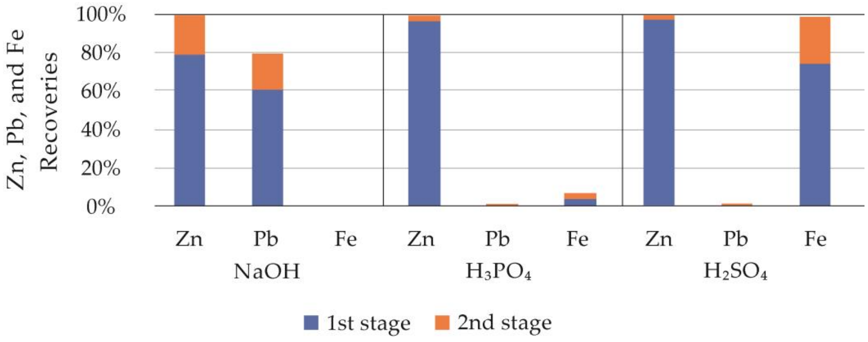

Figure 5. Zinc, lead, and iron recovery from SMD in two stages with sodium hydroxide (240 g L^{-1}—6 M), phosphoric acid (150 g L^{-1}—1.5 M), and sulfuric acid (150 g L^{-1}—1.5 M) at 10% solids and 5 h of agitation per stage at 80 °C.

3.3. Evaluation of Zinc Recovery from Pregnant Solutions by Electrodeposition and Smelting

Preliminary electrodeposition essays, according to the conditions presented in Section 2.3, showed problems when the electrolyte was the pregnant solutions obtained with phosphoric acid leaching. In fact, corrosion of the stainless-steel cathode was observed, as presented in Figure 6. However, there was not a noticeable deposit of metallic zinc. Due to the corrosion observed in the electrodes and the absence of a metallic deposit, no further purification process by cementation was conducted on the pregnant solutions obtained by acid leaching.

Figure 6. Stainless-steel cathode after electrodeposition with phosphoric acid pregnant solution.

Regarding the electrodeposition of pregnant solutions obtained from the alkaline leaching, the preliminary assays were promising. However, due to the presence of Pb and Zn, it was necessary to include a purification stage of cementation with zinc powder to remove these impurities prior to the electrodeposition tests. The analysis of lead content before and after cementation revealed that the process removed 88% of the lead. The purified solutions were then subjected to electrodeposition tests. The combination of graphite anode and stainless-steel cathode showed the best results. In fact, the use of an anode of graphite produced the highest amounts of zinc deposited on the cathode compared to the other two anode materials used (lead and stainless steel).

The chemical analysis of the zinc deposited on the stainless-steel anode was conducted using X-ray diffraction (XRD) and X-ray fluorescence (XRF). The results of the XRF analysis are presented in Table 6. These techniques were employed to evaluate both the crystalline structure and the elemental composition of the deposited zinc. XRD (Table 7) provides insights into the phases present in the deposit, allowing for the identification of any impurities or secondary phases that might have formed during electrodeposition. XRF (Table 6), on the other hand, offers a semi-quantitative analysis of the elemental composition, highlighting the purity of the zinc and the presence of any residual elements such as iron or lead.

Table 6. Semi-quantitative XRF analysis of Zn, Pb, and Fe content in the zinc deposit.

Element	Content (%)
Zn	70.3
Pb	0.1
Fe	0.2

Table 7. X-ray diffraction (XRD) analysis showing the crystalline phases present in the zinc deposit on the stainless-steel anode, identifying any impurities or secondary phases formed during electrodeposition.

Mineral	Content (%)
ZnO	67
Zn	33

As presented in Table 6, the main metallic component of the deposit is zinc, with minimal contamination of the other two metals (Fe and Pb) present in the SMD. These results demonstrate the suitability of the electrolyte for zinc recovery. It is important to mention two keys aspects of the electrolyte. The first aspect is the selectivity of the alkaline leaching towards Zn, minimizing the dissolution of Fe. The second aspect is the effectiveness of the cementation process applied to eliminate Pb from the pregnant solution. The characterization by XRD of the crystalline phases present in the zinc deposit are detailed in Table 7.

Table 7 highlights the presence of metallic zinc on the final deposit. Metallic zinc deposits obtained from electrodeposition are relatively prone to oxidation, which is demonstrated by the high ZnO content (67%). In fact, zinc is more easily oxidized compared to many other metals due to its position in the electrochemical series, where it is listed above metals like copper. This means zinc has a higher tendency to lose electrons and form zinc oxide (ZnO) when exposed to air or moisture. Another factor to consider is the influence of the electrolyte. Using NaOH (sodium hydroxide) as an electrolyte in zinc electrodeposition can indeed influence the oxidation of the zinc deposit. In an alkaline environment, such as one created by NaOH, zinc tends to form a more porous and less compact deposit. This can make the zinc more susceptible to oxidation because the increased surface area and porosity allow for easier access of oxygen and moisture, which promote the formation of zinc oxide (ZnO) on the surface.

The data from Tables 6 and 7 help confirm the quality and purity of the zinc deposit, which is crucial for determining the effectiveness of the electrodeposition process and identifying any necessary adjustments to improve the outcome. Finally, this deposit was purified by smelting, reaching values up to 99% purity. Figure 7 shows the zinc deposit on the stainless-steel cathode after 8 h of electrodeposition with pH = 11.

Figure 7. Zinc deposited on stainless steel cathode after electrodeposition of the pregnant solution obtained by alkaline leaching.

4. Discussion

4.1. Physical, Chemical, and Mineralogical Characterization of SMD

According to Acero [34], if the apparent density of SMD is low (not exceeding 1.5 g cm^{-3}), the dust is more porous, which could complicate its handling and processing as well as negatively affect the leaching efficiency and electrodeposition operations used for metal recovery. In this study, the apparent density obtained for the SMD was 0.8 g cm^{-3}, indicating a potential difficulty for metal recovery by leaching and electrodeposition.

The value of d_{50} was 2.5 μm for the SMD analyzed; comparing this value with those obtained by Lee et al. [35], where a d_{50} of 10.6 μm was reported, the particle size of the SMD used in this work is four times smaller, which could positively influence the leaching speed of zinc but complicate the separation of the pregnant solution at the end of the leaching process.

Regarding the mineralogical composition, the presence of magnetite, zincite, halite, and franklinite was identified, which is consistent with the mineralogical composition of SMD reported by previous studies such as those of Palimakaą et al. and Ricaurte et al. [6,32]. Halite is a compound commonly found in SMD in Ecuador, possibly due to the use of chlorinated fluxes or contamination in scrap metal. The presence of these contaminants has forced smelting companies in that part of Ecuador to purchase imported scrap to achieve better results in steel smelting. The presence of halides like chloride have certain effects on electrodeposition since they increase the conductivity, generate corrosion, and form metallic halides that decrease the efficiency of the metal deposit. The study of this effect is beyond the scope of this study, but it could be incorporated into future studies of zinc refinement by electrodeposition.

4.2. Comparison of Leaching Selectivity of Acid and Alkaline Leaching Agents

Leaching tests using sulfuric acid were used as a starting point for evaluation of the proposed leaching process in two stages. Metal recoveries using this procedure were encouraging. In fact, zinc recoveries of 99.9% were achieved using sulfuric acid as the leaching agent in two stages at 80 °C. Previous studies such as that by Ricaurte et al. [32] reported zinc recoveries of 88% with a concentration of 150 g L^{-1} (1.5 M) during 24 h of agitation without heating. Another study by Halli et al. [15] found that zinc recovery exceeded 80% with 168 h of leaching and an acid concentration of 1.8 M without heating. Therefore, there is an enhancement effect of using the proposed leaching process in two stages with heating. One of the disadvantages of sulfuric acid as the leaching agent is the high recovery of iron in the pregnant solution. For further zinc recovery from the pregnant solution obtained with this agent, the presence of iron as a pollutant of the solution needs to be considered.

Phosphoric acid showed zinc recoveries of 99.6%. Although higher zinc recoveries compared to alkaline leaching were obtained, the resulting pregnant solution contains iron, which is a problem for the subsequent recovery of metallic zinc. It is important to note that phosphoric acid does not leach lead. The use of phosphoric acid proved to be more selective for zinc, while sulfuric acid dissolved all elements present in the SMD. In studies cited in Halli et al. [15], zinc recoveries of less than 10% were achieved under leaching conditions with 10% solids at 20 °C and 168 h of leaching time, using concentrations of 1.92 M and 19.2 M. To improve the separation of zinc and iron, phosphoric acid with di-(2-ethylhexyl) phosphoric acid (D2EHPA) as an organic solvent was used, achieving recoveries of 97% iron and 99% zinc from SMD, according to Azizitorghabeh et al. [36]. Leaching tests were carried out in two stages due to the high presence of zinc in the tailings after the first stage: The second stage, with the same conditions, improved recoveries. The presence of iron or lead in the solutions represents contamination for subsequent processes such as electrodeposition, as it can cause the deposition of these elements on the cathode, resulting in impurities during smelting and lower zinc purity. The use of temperature also contributed to improving zinc recoveries.

Sodium hydroxide appears as the most selective leaching agent for zinc recovery, achieving a recovery value of 99%. This finding is not only supported by the high zinc recovery values but also by the absence of iron in the pregnant solution. Since iron is not leached by sodium hydroxide, this element remains in the tailings, resulting in a pregnant solution that is cleaner and free of iron impurities. Previous studies observed zinc recoveries of only 22% and 25% using sodium hydroxide at concentrations of 12 M and 24 M at 20 °C, with a leaching time of 168 h and a solids percentage of 10% [15]. Consequently, this study clearly demonstrates the beneficial impact of temperature and the implementation of a two-stage leaching process in enhancing zinc recovery, as evidenced by the results presented in Section 3.2.

Another aspect analyzed in the leaching experiments was the solids percentage. The influence of solids percentage on zinc recovery was studied exclusively with sodium hydroxide and not with sulfuric or phosphoric acids due to the selectivity and specificity that NaOH offers in the process. Sodium hydroxide is a highly selective leaching agent that minimizes the dissolution of impurities such as iron, making it a preferred choice for optimizing zinc recovery. Palimakaą et al. [6] conducted leaching experiments using NaOH at various solids percentages (2%, 3%, 5%, and 9%) and observed that the highest recoveries of 88% occurred at 2%, suggesting an inverse relationship between solids percentage and leaching effectiveness. In this study, the influence of solids percentage in a two-stage leaching process is presented, showing a similar trend to that reported by Palimakaą et al. [6].

Additionally, sulfuric and phosphoric acids have different chemical behaviors, where their ability to leach iron might render the effect of solids percentage less relevant or manageable. Therefore, the research focused on NaOH to maximize the purity and industrial relevance of the pregnant solutions.

It is also important to validate the leaching results by the analysis of the solid residue left after the leaching process. The XRF analysis of the solid residues obtained from the first and second leaching stages confirmed the migration of the studied metals from the SMD to the leaching solution. The initial Zn content in SMD was 26.3%, and it decreased to 3.9% after the first leaching stage and further dropped to 0.1% after the second leaching stage when sulfuric acid was used as the leaching agent. In the case of phosphoric acid, a final Zn content of 0.9% was found even after the first leaching stage and remained almost unchanged for the second leaching stage. For sodium hydroxide, a final Zn content of 9% was found after the first stage of leaching and 1% after the second stage of leaching. Iron showed a trend according to the different selectivity observed in the leaching agents studied. Thus, starting from an initial Fe content of 20%, it remained almost unchanged for alkaline leaching (~19%), corroborating the selectivity of NaOH towards Zn over Fe. In the case of phosphoric acid, the final Fe content decreased to 15%, showing limited solubility for Fe in this leaching agent. On the contrary, sulfuric acid leached a significant amount of the Fe in the SMD, giving a final content of 0.9% of iron in the solid residue. Although lead was present in a low concentration (0.9%), there was a decrement in the content of this metal only when alkaline leaching was used (final content of 0.4%), whereas in the acid leaching, the Pb content remained mostly unchanged (~0.8%).

4.3. Evaluation of Zinc Recovery from Pregnant Solutions by Electrodeposition and Smelting

The cementation process was employed to purify the pregnant zinc solutions obtained from the leaching of SMD with sodium hydroxide and phosphoric acid. These leaching solutions contained not only zinc but also impurities such as lead, which could interfere with subsequent electrodeposition. By introducing a more reactive metal, like zinc, into the solution, this cementation process selectively precipitates out these impurities. Specifically, lead can be precipitated as lead hydroxide or lead phosphate. According to the underlying reaction Equation (7) present in the study of Palimakaą et al. [6], using zinc facilitates the removal of more noble metals and significantly reduces lead content. This purification step ensures a cleaner solution, enhancing the efficiency of the electrodeposition process and resulting in a higher-purity zinc product.

$$n_2\mathrm{Me}^{n1+} + n_1\mathrm{M}^0 = n_2\mathrm{Me}^0 + n_1\mathrm{M}^{n}{}_{2}{}^{+} \tag{7}$$

where n_1, n_2 is the ion charge; Me is the cementated metal (Pb); and M is the cementing metal (Zn).

Phosphoric acid is not ideal as an electrolyte for zinc electrodeposition because it can cause oxidation of the stainless-steel cathode instead of facilitating zinc deposition. This is due to several factors: Phosphoric acid is a weak acid with low ionization, resulting in fewer hydrogen ions than necessary for effective metal deposition. It can create an oxidizing environment, especially if impurities like iron are present, leading to corrosion of the cathode. Additionally, the formation of insoluble zinc phosphate compounds can passivate the cathode surface, blocking further zinc deposition. Phosphoric acid's lower ionic conductivity further reduces the efficiency of the electrodeposition process, making it unsuitable for this application.

In the electrodeposition process, using sodium hydroxide as the leaching agent and a pregnant solution previously purified through cementation, zinc was successfully deposited on a stainless-steel electrode, achieving a recovery rate of 99%. Therefore, the study underscores the importance of cementation as an essential purification step preceding electrodeposition. Subsequently, the deposited zinc was smelted, resulting in a product with a purity of 99%.

Electrodeposition results highlighted the importance of electrode material selection, with the graphite anode and stainless-steel cathode combination yielding the best results for zinc deposition and subsequent purification. Graphite is used as an anode because it is chemically inert and resistant to oxidation in alkaline solutions. It does not decompose or react with the electrolyte, minimizing contamination of the solution. On the other hand, stainless steel is suitable as a cathode due to its corrosion resistance and its ability to withstand zinc deposition without degrading, providing an ideal conductive surface for electrodeposition.

The other materials used as anodes presented issues during the experiments. Although lead is seen as stable and less reactive material, the use of an anode made of lead in alkaline conditions was not beneficial due to the potential dissolution of this electrode and its contamination of the electrolyte. Stainless steel can corrode or degrade when used as an anode in concentrated alkaline solutions, which can shorten the anode's lifespan and reduce the efficiency of the electrodeposition process. Additionally, stainless steel is less stable and more reactive under these conditions, which can complicate process control and negatively impact the quality of the coating.

These findings underscore the importance of optimizing leaching conditions, including temperature, agitation time, and leaching agent concentration, to maximize zinc recovery from SMD. The study provides valuable insights for developing efficient and sustainable methods for zinc recovery, contributing to the circular economy and reducing industrial waste.

4.4. Future Insights into the Zinc Recovery from SMD

The primary objective of this study was to conduct a comparative analysis of two leaching agents, namely sodium hydroxide and phosphoric acid, with a focus on their selectivity for zinc (Zn), iron (Fe), and lead (Pb). While this research centers on these aspects, it also identifies additional areas that, although not the main focus, could be explored in future studies on the management of SMD.

Outside the three metals analyzed (Zn, Fe, and Pb), there is some concern in the scientific community regarding cadmium in SMD, as presented by Halli et al. [2]. A cadmium content of about 0.03% was found in the SMD samples analyzed. In previous studies, such as that by Palimakaą et al. [6], a cadmium content of 0.05% was found in steel mill dust. The health problems that cadmium poses during inadequate handling of this residue needs to be considered for proposing the best leaching process. During the leaching process, cadmium and zinc can both dissolve into the solution, making it challenging to selectively recover zinc without also extracting cadmium. According to the leaching experiments conducted in this study, in both cases (alkaline and acid), cadmium was totally leached. Therefore, further investigation in the separation of cadmium from the leaching solution should be addressed in more detailed. However, it is hypothesized that cadmium removal is attained during the cementation process with zinc powder, and this finding could be used as a starting point for future research on zinc recovery from SMD.

In this study, a comparative analysis of the two leaching agents was performed. Further investigation into the redox behavior, the kinetics of each leaching agent, and the formation of intermediate species are interesting aspects for a deeper understanding of the reaction mechanism in each leaching agent, especially in phosphoric acid. Although these aspects are beyond the scope of this study, it is suggested that cyclic voltammetry analysis of the leaching solutions could be a necessary tool to address this issue in a deeper way.

The basis of zinc recovery by electrodeposition is presented in this study. Further investigation to optimize zinc recovery by electrodeposition is beyond the main goal of this study. Certain parameters, such as the kinetics and mechanisms of electrochemical reactions, the diffusion of ions in the electrolyte, and the optimal electrode surface area, should be explored in future studies. One way to obtain these parameters would involve the use of specific techniques like chronoamperometry.

Regarding the leaching agents, an interesting aspect for future research also beyond the main scope of this investigation would be to determine the corrosion caused by the leaching agent on exposed surfaces after the leaching experiments. One method suggested to perform this analysis is energy dispersive X-ray spectroscopy (EDS). Another research direction that should be investigated is the recyclability of the leaching agents. Given the use of strong alkalis and acids in the leaching process, it is essential to develop a purification method to remove impurities, enabling continuous recirculation of the leaching agents.

5. Conclusions

The SMD produced by an electric arc furnace in an Ecuadorian facility was analyzed, revealing a particle size of 5.4 µm (d_{80}). The primary constituents identified included magnetite (Fe_3O_4) at 42%, zincite (ZnO) at 38%, halite (NaCl) at 8%, and franklinite or zinc ferrite ($ZnFe_2O_4$) at 4%. The characteristics of this residue highlight the need for developing an efficient zinc recovery process.

Two-stage acid and alkaline leaching tests were conducted at 80 °C for 5 h per stage. Phosphoric acid and sodium hydroxide were used as leaching agents, with additional tests using sulfuric acid as a reference. Leaching of the SMD with sulfuric acid (150 g L^{-1}—1.5 M) achieved zinc recoveries of 97% in the first stage and 2.9% in the second stage, resulting in a total zinc recovery of 99.9%. With phosphoric acid (150 g L^{-1}—1.5 M), recoveries were 96.5% in the first stage and 3.1% in the second stage, resulting in a total zinc recovery of 99.6%. Leaching with sodium hydroxide (240 g L^{-1}—6 M) resulted in zinc recoveries of 79.2% in the first stage and 20% in the second stage, resulting in a total zinc recovery of 99.2%. Maintaining a heating temperature of at least 80 °C and employing a two-stage leaching process were crucial for achieving better zinc recoveries with both acids and bases.

Phosphoric acid is more selective for zinc compared to sulfuric acid, which dissolves all elements present in the SMD (iron, zinc, and lead). However, phosphoric acid still dissolves a small amount of iron (approximately 7%), whereas alkaline leaching results in minimal iron dissolution (less than 0.01%). Sodium hydroxide is highly selective for zinc, leaving iron in the tailings and subsequently removing iron and lead from the solution through cementation. The choice of leaching agent significantly impacts the efficiency of the zinc recovery process.

The influence of solid percentage in the leaching process was also explored. During leaching with sodium hydroxide, the optimal condition for zinc recovery was found to be maintaining a solids concentration of 10% compared to the higher solid percentages tested (20%, 30%, and 40% solids). The 10% solids concentration allowed for more efficient contact between the leaching agent and the metal particles, enhancing the dissolution process and maximizing zinc extraction. This optimized leaching condition ensured higher zinc recovery rates and a more effective leaching process overall. Similar results were obtained in preliminary leaching experiments with phosphoric acid.

The cementation process effectively served as a purification stage for the NaOH-pregnant solution, achieving an 80% reduction in lead content. The purified solution constituted a significantly cleaner solution for the subsequent electrodeposition.

The electrodeposition of zinc from alkaline leaching using a graphite anode and stainless-steel cathode yielded the best results, with higher amounts of zinc deposited on the cathode. This zinc deposit can be purified by smelting to achieve up to 99% purity. The use of lead and stainless steel as anodes was also tested, but the results were unsatisfactory.

Electrodeposition using phosphoric acid was tested with a graphite cathode and a stainless-steel anode, but the results were poor, as the stainless-steel electrode corroded. Therefore, it is recommended to conduct further tests with alternative cathode materials to those used in this study.

This comparative study on the selectivity of leaching agents—sodium hydroxide and phosphoric acid—highlights the effectiveness of a two-stage leaching process with heating, achieving nearly 100% zinc recovery from steel mill dust. The findings demonstrate that sodium hydroxide is the most selective and efficient leaching agent for zinc recovery, surpassing phosphoric acid in performance. The study contributes valuable insights into optimizing hydrometallurgical processes for the treatment of steel mill dust, emphasizing the importance of selecting appropriate leaching agents to maximize metal recovery.

The findings of this comparative study can be applied to the Ecuadorian steel industry, where SMD is not currently managed or processed domestically. Instead, these residues are typically exported. By integrating the methods and insights from this research, Ecuador could establish an efficient recovery process for zinc and other metals from steel mill dusts, adding value within the local industry.

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