

# INNOVATIVE HYDROMETALLURGY FOR GALVANIC SLUDGE SUSTAINABLE RECOVERY

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**Abstract:** The manuscript explores the feasibility of recovering zinc and iron from waste galvanic sludge generated during galvanic plating processes. Galvanic sludge, characterized by elevated concentrations of heavy metals, represents a suitable candidate for hydrometallurgical recycling. The primary objective of the experimental and practical phases was to extract zinc and iron through the leaching of galvanic sludge. Leaching procedures were conducted using sulfuric acid at varying temperatures and time durations, augmented by the introduction of oxidizing agents such as hydrogen peroxide or ozone. Subsequent separation of the leach and filtrate was achieved through filtration. The leachate underwent additional processing involving the precipitation of iron and other metals, employing diverse agents. Following further filtration, electrolysis was employed to attain pure zinc on the cathode, utilizing an electrical voltage of approximately 3 V. Comprehensive chemical analyses were conducted on all intermediate products, including the leachate, leach liquor, filtrate, solid precipitate, and the separated metal on the cathode. The outcomes of these analyses are meticulously presented in tables and graphs.

**Keywords:** metals; galvanic sludge; leaching; zinc; iron; electrolysis

## 1. INTRODUCTION

Innovation stands as the cornerstone of progress across diverse scientific and industrial domains. Its significance lies in its transformative capacity to revolutionize existing paradigms, foster breakthroughs, and propel societies toward a more advanced and sustainable future. In the realm of science, innovation acts as the catalyst for pushing the boundaries of knowledge, continually expanding our understanding of the natural world (Jagielska-Wiaderek and Klimecka-Tatar, 2023; Dziuba and Ingaldi, 2015, Grebski, Mazur, 2022).

In the broader context, innovation is not merely a luxury but a necessity, particularly in addressing contemporary challenges. It serves as a driving force behind economic

growth, technological evolution, and societal well-being. The relentless pursuit of innovative solutions is crucial for overcoming complex problems, ranging from environmental sustainability and resource management to healthcare and beyond (Larina et al., 2021; Krynke, 2021; Kuzior, 2022; Ulewicz et al. 2013).

Innovation is characterized by a dynamic interplay between creativity, research, and application. It involves the development and implementation of novel ideas, processes, and technologies that redefine the status quo. This dynamic cycle of discovery and implementation not only propels scientific disciplines forward but also fuels industrial progress, driving improvements in efficiency, productivity, and overall quality of life (Mazur, 2018; Muneer et al., 2023; Walichnowska and Idzikowski, 2023; Kuzior and Zozul'ak, 2019).

As we navigate an era marked by rapid technological advancement and global interconnectedness, the importance of fostering a culture of innovation cannot be overstated. It is through innovation that societies can address pressing issues, adapt to changing circumstances, and forge a path toward a more resilient and sustainable future. In essence, innovation is the cornerstone of human progress, providing the means to navigate the complexities of the modern world and unlock the untapped potential of the future (Škúrková and Ingaldi, 2014; Lipiński and Ulewicz, 2023; Krynke et al., 2022; Borkowski et al. 2012).

Innovation plays a pivotal role in advancing the field of hydrometallurgy, ushering in a new era of scientific exploration and technological breakthroughs. The imperative for innovation in hydrometallurgy stems from the ever-growing demand for sustainable and efficient methods of extracting metals from ores. As global industrial and technological landscapes evolve, the need for environmentally friendly and economically viable metallurgical processes becomes increasingly critical.

The dynamic nature of hydrometallurgy, which involves the use of aqueous solutions for extracting and recovering metals, necessitates constant innovation to address challenges and optimize existing methodologies. Innovations in this field contribute not only to enhanced resource efficiency but also to the reduction of environmental impact associated with traditional metallurgical practices.

Hydrometallurgical processes have garnered considerable attention in research due to their ability to process complex ores, concentrates, and wastes while mitigating environmental pollution (Dvořák and Jandová, 2005; Radzimska-Lenarcik et al. 2018 and 2020). The recovery of waste materials and metals from diverse secondary sources is crucial for both economic and environmental reasons. Zinc, renowned for its versatility, finds extensive applications in plating, coating, and alloying with other metals (Barakat, et al., 2007). The burgeoning demand for zinc has prompted industries to explore secondary sources like zinc waste, zinc slag, spent batteries (Zn–C, Zn–Mn, Zn–Ag<sub>2</sub>O) and leaching residues as potential resources (Sokić et al., 2019, Radzimska-Lenarcik and Ulewicz, 2019, Radzimska-Lenarcik et al. 2020a).

Empirical data gathered from the performance of galvanized steel in various environments underscore zinc's effectiveness in preventing corrosion, surpassing other surface treatments with its significantly slower corrosion rate compared to iron (Brožová, et al., 2021). Hydrometallurgical methods described in the literature for the processing of solid and liquid waste generated during galvanizing and galvanization (e.g. leaching, ion separation, removal from aqueous solutions) (Křištofová, 2001; Brožová et al., 2016; Havlík et al., 2016; Gupta, 2003), provide viable avenues for treating zinc sludge. Existing

studies predominantly focus on the leaching method using sulfuric (Křištofová, 2001) or hydrochloric acid (Gupta, 2003) and for separation zinc ions from other metal ions (Ulewicz and Radzaminska-Lenarcik, 2012 and 2014).

This paper presents preliminary findings from studies on the hydrometallurgical treatment of galvanic sludge from industrial sources. Presently managed domestically by zinc producers or sold to foreign recyclers, this waste stream is a focal point for our investigation. The research objectives encompass determining the impact of solid-to-liquid ratio, agitation rate, and temperature on zinc leaching efficiency with sulfuric acid, incorporating oxidizing agents such as hydrogen peroxide and ozone. These findings aim to contribute to the development of an enhanced process for recycling zinc and iron for use as secondary raw materials (Havlík et al., 2016; Gupta, 2003; Gacayak et al., 2014; Ahn et al., 2019; Jackson, 1986).

Ore raw materials stand as a primary source for metal production, necessitating processing through treatment procedures to yield a concentrate serving as the foundation for specific metal production. Various methods, including pyrometallurgy, hydrometallurgy, and electrometallurgy, are employed in metal production, followed by refining processes (Brožová, et al., 2021).

The exploration of innovative technological approaches for processing polymetallic ores and concentrates with unconventional non-ferrous metal content, while adhering to environmental standards, is a contemporary challenge in metallurgical mining. The use of pyrometallurgical processes raises environmental concerns due to the substantial production of sulfur dioxide (SO<sub>2</sub>) (Lisińska, et al. 2018). In response, recent years have witnessed a surge in research on hydrometallurgical processes, given their capacity to handle complex ores, concentrates, and wastes while concurrently mitigating environmental pollution.

The fundamental sequence in the hydrometallurgical production of metals involves leaching pre-treated enriched ore, typically subjected to roasting to generate highly soluble sulfates. The resulting product comprises two phases: the leach solid residue and the leach solution, the latter containing the sought-after metal. Metal recovery from the leachate involves several processes. In cases where the feedstock is relatively rich, multiple metals may still be present in the leachate. These can undergo further leaching with specialized reagents, producing additional leachate and a depleted waste stream. This intricate process contributes to the efficient extraction of metals while addressing environmental concerns associated with traditional methods.

The primary goal of this work is to extract zinc and iron through the leaching of galvanic sludge. A secondary objective involves a theoretical analysis of hydrometallurgical processes. The practical phase involves sample treatment and subsequent leaching of waste sludge using sulfuric acid concentrations of 10%, 20%, and 30%. The thesis culminates in an assessment of conditions yielding the highest zinc and iron yields post-leaching, accompanied by recommendations for subsequent procedures.

## **2. MATERIAL AND METHOD**

The objective of the experimental phase was to validate optimal leaching conditions for extracting zinc and iron, essential metals of interest, from waste galvanic sludge. The initial treatment involved drying and crushing the sludge sample to achieve the desired particle size. Subsequently, a leaching process was conducted using sulfuric acid and nitric acid as selected leaching agents. This choice was informed by an extensive review

of literature sources focusing on the leaching of waste materials. Leaching solutions were prepared from concentrated acids, diluted to a 20% solution. Additional leaching conditions were set at 40°C and 60°C for a duration of 6 hours, with the incorporation of oxidizing agents, namely hydrogen peroxide and ozone.

The subsequent phase of the experimental work aimed to identify the optimal conditions for maximizing the yield of zinc and iron from the waste galvanic sludge. Sulfuric acid was chosen for this particular experiment, with concentrations of 10% and 30%. Other conditions remained constant, including the use of hydrogen peroxide and ozone as oxidizing agents. The chemical analysis of the sample is outlined in Table 1.

Table 1

Chemical composition of input galvanic sludge of two galvanic plants – average from three analyses (wt.%)

Source	Zn	Fe	Cr	Mn	Cu	Co	Cl	S
Plant D	11.3	7.1	0.1	0.08	0.1	-	5.1	0.5
Plant H	15.8	2.7	0.5	0.03	-	0.23	2.4	0.1

The input samples of the waste galvanic sludge underwent analysis for zinc and iron content using the ED-XRF method in the laboratories of the Faculty of Materials and Technology. Prior to analysis, the waste sludge samples underwent pretreatment involving drying under normal conditions and manual grinding to achieve the required particle size, as illustrated in Figure 1.



Fig. 1. Preparation of samples for leaching and the leaching process

Two acids, sulfuric and nitric, were selected for the hydrometallurgical leaching method based on an analysis of relevant literature. Leaching solutions were prepared from concentrated acids, with a dilution to a 20% solution. In the subsequent series of experiments, sulfuric acid was chosen in two dilutions, 10% and 30%, aiming to assess the impact of leaching solution concentration on the recovery of zinc and iron in the leachate. Another variable introduced in the experiments was the addition of oxidizing agents in the form of  $H_2O_2$  and ozone ( $O_3$ ), at temperatures of 40 and 60°C for a duration of 6 hours. This systematic approach in the experimental design aimed to comprehensively explore the interplay of leaching conditions and their influence on metal recovery from galvanic sludge.

### 3. RESULTS

The receptacles accommodating the meticulously prepared samples (200 ml of sulfuric acid, amalgamated with 50 g of the precisely measured specimen, adhering to a 1:4 ratio)

were positioned upon electromagnetic stirrers. The requisite temperature, precisely set at 50°C, was individually configured for each electromagnetic stirrer. Subsequent to the stipulated duration of time (2 and 5 hours), the leaching process was duly terminated, and the residual leachate and leaching residue were meticulously partitioned through the process of filtration.

The post-leaching samples underwent a meticulous chemical analysis to determine the concentrations of zinc and iron. Figure 2 delineates the analytical outcomes for the leachate, encapsulating zinc and iron, derived from samples sourced from galvanizing plants D and H subsequent to the leaching procedure conducted in a 20% H<sub>2</sub>SO<sub>4</sub> solution at 50°C, following two- and five-hour durations. The graphical representation underscores a more subdued reduction in zinc content within the leachate, juxtaposed against a more pronounced decline in iron content over the elapsed duration.

The subsequent phase of the experimental investigation involved the utilization of prepared samples within a 200 ml beaker containing sulfuric acid solutions with concentrations of 10% and 30%, concomitantly with 50 g of a meticulously measured specimen of waste electroplating sludge, maintaining a ratio of 1:4. These assemblies were subjected to electromagnetic stirrers, with distinct temperatures (40°C and 60°C) individually set for each apparatus. Following a predetermined duration of 6 hours, the leaching process was curtailed, and the resultant leachate and solid leaching residue underwent meticulous separation via filtration. The leachate was systematically collected upon the culmination of the 6-hour experiment. Hydrogen peroxide and ozone were incorporated as oxidizing agents in these experimental iterations.

Subsequent to the leaching procedures, the obtained samples underwent comprehensive chemical analyses to ascertain the concentrations of zinc and iron, as presented in Table 3.

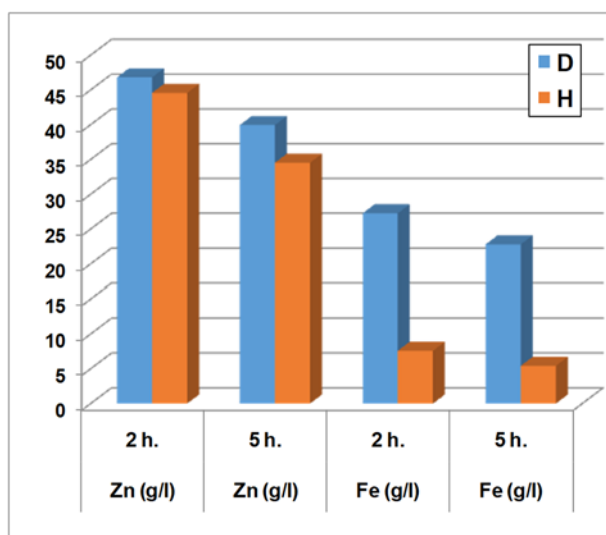


Fig. 2. The resulting content of zinc and iron content in the leach (galvanic plants D and H)

Table 2 delineates that an elevated concentration of sulfuric acid, coupled with increased temperature, precipitates a more thorough dissolution of both zinc (Zn) and iron (Fe). Nonetheless, it is noteworthy that over time, the concentrations of both Zn and Fe exhibit a diminishing trend. Notably, the introduction of hydrogen peroxide during the leaching process demonstrated heightened efficacy in a 30% H<sub>2</sub>SO<sub>4</sub> environment compared to

ozone. Conversely, ozone manifested greater reactivity during leaching in a 10% H<sub>2</sub>SO<sub>4</sub> milieu. Irrespective of the sulfuric acid concentration, it is discerned that the oxidation agent exerts a positive influence on the leaching process.

Table 2

The resulting content of zinc and iron in the H<sub>2</sub>SO<sub>4</sub> leachate. Source: H

40°C, 10% H <sub>2</sub> SO <sub>4</sub>			60°C, 10% H <sub>2</sub> SO <sub>4</sub>		
Addition, time	Zn [mg·l <sup>-1</sup> ]	Fe [mg·l <sup>-1</sup> ]	Addition, time	Zn [mg·l <sup>-1</sup> ]	Fe [mg·l <sup>-1</sup> ]
H <sub>2</sub> O <sub>2</sub> , 2 h	10,800	0.805	H <sub>2</sub> O <sub>2</sub> , 2 h	16,400	4.52
H <sub>2</sub> O <sub>2</sub> , 4 h	11,100	2.77	H <sub>2</sub> O <sub>2</sub> , 4 h	24,400	15.8
H <sub>2</sub> O <sub>2</sub> , 6 h	11,400	5	H <sub>2</sub> O <sub>2</sub> , 6 h	36,100	18.6
O <sub>3</sub> , 2 h	18,600	4.7	O <sub>3</sub> , 2 h	17,300	344
O <sub>3</sub> , 4 h	19,900	75.7	O <sub>3</sub> , 4 h	22,300	820
O <sub>3</sub> , 6 h	20,000	2,150	O <sub>3</sub> , 6 h	23,200	4360
40°C, 30% H <sub>2</sub> SO <sub>4</sub>			60°C, 30% H <sub>2</sub> SO <sub>4</sub>		
Addition, time	Zn [mg·l <sup>-1</sup> ]	Fe [mg·l <sup>-1</sup> ]	Addition, time	Zn [mg·l <sup>-1</sup> ]	Fe [mg·l <sup>-1</sup> ]
H <sub>2</sub> O <sub>2</sub> , 2 h	40,200	6,980	H <sub>2</sub> O <sub>2</sub> , 2 h	47,500	8,480
H <sub>2</sub> O <sub>2</sub> , 4 h	31,600	5,781	H <sub>2</sub> O <sub>2</sub> , 4 h	35,400	6,890
H <sub>2</sub> O <sub>2</sub> , 6 h	31,700	6,200	H <sub>2</sub> O <sub>2</sub> , 6 h	43,700	8,420
O <sub>3</sub> , 2 h	29,700	5,140	O <sub>3</sub> , 2 h	32,400	7,260
O <sub>3</sub> , 4 h	19,500	3,660	O <sub>3</sub> , 4 h	27,920	5,200
O <sub>3</sub> , 6 h	15,600	4,000	O <sub>3</sub> , 6 h	13,700	4,360

Figure 3 provides a comparative illustration of the outcomes derived from ICP-AES (inductively coupled plasma atomic emission spectroscopy) analyses conducted on the initial sludge, the leaching residue subsequent to leaching in 10% H<sub>2</sub>SO<sub>4</sub>, the leaching process with the addition of hydrogen peroxide in 10% H<sub>2</sub>SO<sub>4</sub>, and the leaching process in the presence of ozone in 30% H<sub>2</sub>SO<sub>4</sub>. Notably, leaching in 10% H<sub>2</sub>SO<sub>4</sub> resulted in an approximate 40% reduction in zinc content within the leach liquor, coupled with the precipitation of iron (Fe). The introduction of hydrogen peroxide or ozone exhibited a favorable impact on the reduction of both Fe and Zn concentrations in the leach liquor.

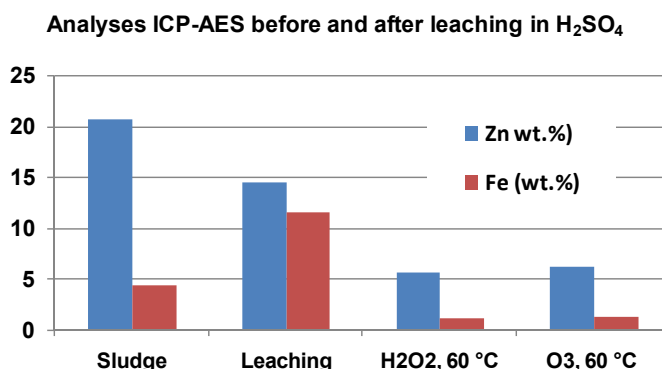


Fig. 3. The resulting of zinc and iron content in the leaching residue (galvanic plant H2)

Additional elements, namely Cr, Mn, Co, Ni, and Cu, were identified in the leach liquor, as illustrated in Figure 4. This observation suggests that their effective separation occurs

during the leaching process in a 30% H<sub>2</sub>S<sub>4</sub> solution at a temperature of 60°C, particularly in the presence of oxidation agents.

The analyses conducted indicate that both the leach and the leach liquor harbor elements such as Zn, Fe, Cr, Mn, and Co, which necessitate prior separation before initiating the electrolysis process.

Subsequently, the next phase involved the precipitation of iron and potentially other metals from the leach after the initial filtration. To achieve this, precipitation utilizing NH<sub>4</sub>OH ammonia was initiated. Specifically, 200 g of H sample sludge underwent leaching in 1 liter of 20% H<sub>2</sub>SO<sub>4</sub>. Following this, the leach was meticulously separated from the indissoluble fractions via filtration, resulting in a filtrate volume of 920 ml. Subsequently, the solution underwent a secondary filtration process to further isolate precipitated Fe(OH)<sub>3</sub> and other solid byproducts. The pH of the solution was maintained at 7.5.

Prior to commencing electrolysis, a sample was extracted from the solution for subsequent analyses. The solution was then transferred to a vessel, wherein two electrodes were immersed. A lead plate served as the anode, while an aluminum plate functioned as the cathode. The solution was subjected to electrolysis under a voltage of 3V and a current of 0.5A. Following the electrolysis, both the liquid and solid products underwent thorough analysis.

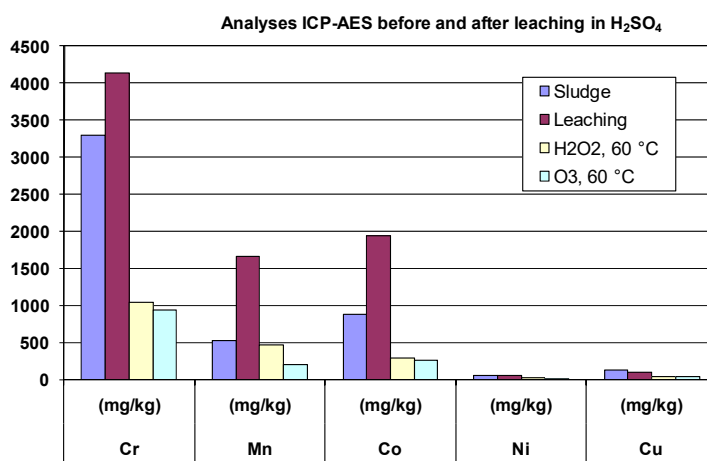


Fig. 4 Content of Cr, Mn, Co, Ni and Cu in the leaching residue (galvanic plant H2)

Table 3

Results of chemical analyses of solution in sulfuric acid, before and after electrolysis [mg·l<sup>-1</sup>]

Source	Conditions	Method	Zn	Fe	Cr	Cu	Mn	Ni
D3	Leaching 20% H <sub>2</sub> SO <sub>4</sub> , 50°C, 4 h	ICP-AES	15,450	10,600	95.5	36.6	119.5	7.1
		AAS	15,000	12,800	67.1	38.9	141.0	11.1
H3	Leaching 20% H <sub>2</sub> SO <sub>4</sub> , 50°C, 4 h	ICP-AES	29,250	5,150	885	35.7	56.5	8.6
		AAS	27,600	5,810	516	34.8	586	12.0
D3	Before electrolysis	AAS	3,290	<0.1	<0.1	5.08	2.33	1.35
	After electrolysis		2,100	<0.1	<0.1	1.65	1.97	1.37
H3	Before electrolysis		6,990	<0.1	<0.1	1.03	31.8	1.33
	After electrolysis		4,930	<0.1	<0.1	<0.1	0.3	0.60

Table 3 presents the analytical outcomes of liquid samples subsequent to leaching and solutions before and after electrolysis. Samples D3 and H3, characterized by chemical compositions outlined in Table 1, underwent leaching in 20% H<sub>2</sub>SO<sub>4</sub> at 50°C for a duration of 4 hours. Notably, the Zn concentration in the leachate for sample H3 was double compared to D3, while the reverse was observed for Fe, aligning with the elemental content of the initial sludge. Additionally, the presence of Cr, Mn, Cu, and Ni was discerned in the leachate.

Following leaching, the leachates underwent precipitation using a 25% solution of NH<sub>4</sub>OH to eliminate Fe, during which Fe(OH)<sub>3</sub> and potentially hydroxides of other metals formed. The resulting solid precipitate was separated from the filtrate through filtration. The filtrate was subjected to analysis both before and after electrolysis, as depicted in Table 3. It is evident from the table that the concentrations of Cu, Mn, and Ni substantially decreased compared to their levels in the leach liquor. Notably, Fe and Cr were absent in the solutions after electrolysis. The diminished Zn concentration both before and after electrolysis can be attributed to the filtrate's acidity, which was excessively high due to the concentrated H<sub>2</sub>SO<sub>4</sub> content.

Table 4

Results of chemical analyses of leaching residues, coagulants and metals after electrolysis [wt.%]

Source	Condi-tions	Method	Zn	Fe	Cr	Cu	Mn	Ni	Pb
D3	Leaching residue	ICP-AES	3.00	2.20	0.025	0.0066	0.031	0.0013	0.0071
		ED-XRF	2.70	1.92		0.0105	0.0230	0.0015	
H3	after leaching	ICP-AES	6.45	1.21	0.14	0.0054	0.0085	0.0012	
		ED-XRF	3.75	0.68	0.07	0.0082	0.008	0.0034	
D3	Coagulating with NH <sub>4</sub> OH	ICP-AES	5.31	16.4	0.17	0.010	0.20	0.0021	0.0019
		ED-XRF	3.67	9.6	0.09	0.018	0.12	0.0030	0.0020
H3	Coagulating with KOH	ICP-AES	3.1	14.8	2.57	0.0063	0.08	0.013	
		ED-XRF	2.57	11.6	1.89	0.04	0.11		
D3	Metal on the cathode	ICP-AES	80	0.029	0.0005	0.62	0.0012	0.09	0.015
		ED-XRF	61	0.080	0.05	1.07		0.10	0.04
H3	Metal on the cathode (KOH)	ICP-AES	67	0.0008	0.0003	0.20	0.0008	0.050	0.057
		ED-XRF	67.2	0.45	0.06	0.45		0.07	0.08
		ED-XRF	63.9	0.41	0.0003	0.79	0.024	0.037	0.033

Table 4 provides the outcomes of chemical analyses conducted on solid samples, encompassing leaching residues and coagulates subsequent to ammonia precipitation,



along with the metallic content obtained on the cathode. Two distinct analytical methods, namely ICP-AES and ED-XRF, were employed.

For both sludge samples, the leaching residue exhibited a notably diminished content of Zn, ranging from approximately 3 to 6 wt.%, and Fe, falling below 2 wt.%. Other elements were observed at levels ranging from tens to hundreds of ppm. Following precipitation using ammonia, Fe, and other heavy non-ferrous metals were conspicuously concentrated in the precipitate. Positive effects were also observed with the use of KOH for precipitation. However, the utilization of  $\text{Na}_2\text{CO}_3$  did not yield favorable results.

Electrolysis conducted at a voltage of 3V, a current ranging from 1 to 1.2A, and a specified electrolysis duration resulted in a notable accumulation of zinc on the cathode. Furthermore, a substantial presence of copper (0.5 to 1 wt.%) was identified, while other metallic elements were detected at levels ranging from units to tens of ppm.

#### 4. CONCLUSIONS

This work aimed to develop a hydrometallurgical method for extracting metals from waste galvanic sludge, specifically focusing on identifying optimum leaching conditions for sludge with high Zn and Fe content. The waste sludge samples underwent treatment and leaching in sulfuric acid solution under predefined conditions. The leaching process product and solutions were analyzed using ED-XRF and ICP-AES, respectively.

Sulfuric acid was applied at various temperatures and leaching times, potentially incorporating hydrogen peroxide or ozone. Notably, the oxidizing agent ozone emerged as the most effective. However, the recovery of metals from electroplating sludge using inorganic acids remains constrained, as acids alone, devoid of other oxidizing agents, exhibited subpar leaching properties. Consequently, the ongoing research emphasizes the selection of process parameters to enhance the concentration of zinc and iron, predominant elements in electroplating sludge.

The proposed leach processing involved the precipitation of iron and other metals (Cr, Mn, Cu, Co, Ni...) using ammonia, potentially KOH, proving to be a judicious approach. The resulting filtrate, post-precipitation, underwent electrolysis in the acidic environment of  $\text{H}_2\text{SO}_4$ . The electrolysis yielded high-purity zinc separation on the cathode, with minimal contamination by copper.

In the subsequent phase of the research, optimization of the entire galvanic sludge processing is imperative to obtain zinc, possibly iron, of the required purity. This stage focuses on achieving maximum zinc concentrations in a form suitable for subsequent refining processes, particularly through distillation.

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