



Spongy Zinc Powder Production from Cold Filter Cake to Use in Cold Purification Stage

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ABSTRACT

The hydrometallurgical extraction of zinc often involves a crucial cold purification stage to remove impurities such as cadmium, cobalt, and nickel from the zinc sulfate electrolyte. The efficiency of this purification hinges on the availability of high-quality zinc dust as a cementation agent. A cost-effective approach to procuring this vital reactant involves processing cold filter cake (CFC), a byproduct of the leaching and purification processes. This study focuses on the production of zinc powder from CFC and its application in the cold purification stage of impure zinc sulfate solutions. The research begins with the alkaline leaching of CFC using a 7 molar NaOH solution at a temperature of 75 °C for 2 h. During the leaching process, 84 % of zinc was dissolved while keeping cadmium and nickel mostly in the residue. The filtrate from the leaching process was then used as an electrolyte in an electrowinning process to produce spongy zinc powder with around 82 % current efficiency. Subsequently, the cold purification of the industrial zinc sulfate solution is performed using spongy zinc powder. The solution is heated to 50 °C and agitated at 300 rpm. A pre-determined quantity of spongy zinc powder is introduced to facilitate the cementation of impurities, specifically cadmium and nickel, which are present at concentrations of 60 and 250 mg.L⁻¹, respectively. The purification process is optimized by adjusting the zinc powder dosage to 5 g, resulting in cadmium and nickel concentrations reduced to 1.4 and 1.5 mg.L⁻¹, respectively, thus meeting the stringent requirements for electrowinning.

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1. Introduction

Zinc production and recovery from secondary sources are increasingly vital due to the finite nature of primary zinc resources and the growing demand for sustainable practices. Secondary zinc, primarily sourced from scrap materials like galvanized steel and metallurgical waste, can be effectively recovered through various innovative methods. Zinc is predominantly extracted from sulfide ores through the hydrometallurgical process, accounting for approximately 80 % of global production. This method, favored for its efficiency and reduced environmental impact compared to pyrometallurgy, involves a multi-stage process beginning with ore concentration via froth flotation to yield a zinc concentrate. The concentrate then undergoes roasting at high temperatures, converting zinc sulfide to zinc oxide and releasing sulfur dioxide. Critically, the captured sulfur dioxide is converted into sulfuric acid, a key reagent in subsequent leaching stages. Leaching, conducted in dilute sulfuric acid, dissolves the zinc oxide, generating a zinc sulfate solution. This process is typically conducted in two stages: a neutral leach targeting zinc oxide and a hot acid leach to break down zinc ferrites. The resulting zinc sulfate solution invariably contains impurities, necessitating rigorous purification. Iron is removed through oxidation and precipitation, like jarosite, goethite, or hematite. Subsequently, zinc dust cementation is employed to displace copper, cadmium, cobalt, and nickel in two cold and hot purification stages. The purified zinc sulfate solution is then subjected to electrowinning, where zinc ions are reduced at the cathode, yielding high-purity zinc [1-4].

However, the hydrometallurgical process is not without its environmental challenges. Significant waste streams are generated, including roasting residue, iron precipitation residues (most notably jarosite), purification residues such as hot and cold filter cakes, and electrolyte bleed. Jarosite, containing hazardous traces of heavy metals, requires careful management through secure landfills or stabilization/solidification techniques. Purification residues, rich in zinc and other valuable metals, are often reprocessed for metal recovery [3-10].

Zinc recovery from cold filter cake (CFC) is a significant area of research, particularly due to the environmental implications of managing hazardous waste. The CFC, a byproduct of the zinc plant, contains valuable metals such as cadmium, nickel, and zinc, which can be recovered through various leaching and extraction processes. The leaching process typically involves using aqueous solutions, such as sulfuric acid, to dissolve metals from the CFC. This method is effective for extracting zinc and other metals like cadmium and nickel. Filter cakes from zinc processing contain toxic heavy metals, which pose environmental risks if not properly managed. Leaching processes must be designed to minimize environmental impact and ensure safe disposal or reuse of the treated residues. Research focuses on optimizing leaching conditions to selectively extract zinc while minimizing the extraction of other metals. This selective approach helps in improving the purity of the zinc product and reducing waste. Studies have explored the use of additives to stabilize filter cakes and reduce the leachability of heavy metals. However, the focus on zinc leaching specifically may require different strategies to enhance zinc extraction efficiency and selectivity [11-15]. Behnajady and Moghadam [16] focused on selective zinc leaching from hazardous As-bearing zinc plant purification filter cakes using pH-dependent tests and optimal conditions, achieving nearly 95 % Zn extraction while minimizing impurities, specifically under conditions of 333 K, 120 min, pH 5, and 150 g/L. Balesini et al. [17] leached zinc from the CFC using sulfuric acid to extract zinc, and then utilized D2EHPA for solvent extraction. This study achieved 98.8 % zinc recovery under optimized conditions of 40 vol.% extractant, pH 2.5-3, and a 4:1 phase ratio. Sombhatla et al. [18] used ammoniacal carbonate leaching. This method has been validated for cold purification of cake, which contains 10-20 % zinc. The leaching process is influenced by parameters such as temperature, agitation, and solvent concentration, with successful implementation in commercial settings. Ashtari and Pourghahramani [19] applied sodium hydroxide to alkali leach zinc from a hot purification filter cake. Research indicates that NaOH concentrations of 3 to 9 M can significantly impact zinc extraction from hot filter cake, achieving up to 83.4 % recovery under optimal conditions.

In this study, a novel and practical process is proposed for the recovery and reuse of zinc from CFC, a hazardous industrial residue. The innovation lies in the combined application of selective alkaline leaching and direct electrowinning to produce spongy zinc powder, which is subsequently utilized in the cold purification stage of industrial zinc sulfate solutions. While previous studies have separately addressed zinc recovery from residues or purification using commercial zinc dust, the present work demonstrates, for the first time, the complete integration of CFC treatment and zinc powder utilization within the same process chain. The results confirm that the in-house produced spongy zinc powder effectively reduces cadmium and nickel concentrations to acceptable levels for electrowinning, highlighting both the technical feasibility and industrial relevance of this approach.

2. Materials and Methods

2.1. Material

Sodium hydroxide (NaOH) was purchased from Mojallali Co. The main experimental material was CFC, containing approximately 19.5 % zinc, 21.5 % cadmium, and 4.5 % nickel. The zinc sulfate solution's pH was 3.5 and had 70 g/L zinc. The zinc sulfate solution, cadmium, and nickel impurities were 60 and 250 mg.L⁻¹, respectively. Both the CFC and the zinc sulphate solution were provided by Zanjan Zinc Khales Sazan Industries Company (ZZKICo) in Zanjan, Iran.

2.2. Experimental Procedure

The flowchart is shown in Fig. 1 with details of the experiment. The alkaline leaching of zinc was done in a 5 L Erlenmeyer flask with the following conditions: NaOH concentration 7 M, pulp density 100 g/L, reaction temperature 75 °C, and reaction duration 2 h. The process begins by adding 400 g CFC to a 4 L 7 M NaOH solution and heating the pulp to 75 °C. The solution was continuously stirred at 500 rpm using a mechanical stirrer to ensure uniform mixing and enhance the leaching process. Finally, the leaching slurry was filtered immediately, and the filtrate was analyzed by atomic absorption spectroscopy, Varian 240AA. 3.5 L of alkaline leaching filtrate was used as an electrolyte in electrowinning to produce zinc powder. The electrolysis of the alkaline solution, conducted using two stainless steel sheets of dimensions 12×9 cm² units serving as electrodes, resulted in an average applied voltage of 3.3 V and an average current of 5 A at ambient temperature for 10 h.

The cold purification of industrial zinc sulfate solutions is a critical step in the refinement of zinc. Experiments were conducted utilizing spongy zinc powder in a 2-liter beaker to achieve this purification. The procedure involved introducing 1 liter of impure zinc sulfate solution into the beaker, which was then placed on a heater and agitated at 300 rpm. The solution temperature was elevated to 50 °C, and the pH, measured at approximately 3.5, was deemed appropriate for initiating the purification process. Following this, a pre-determined quantity of spongy zinc powder was added, facilitating the cementation of impurities by the metallic zinc under continuous stirring. Once the designated purification time had elapsed, the solution was filtered. The concentration of nickel and cadmium after cold purification was measured in the resulting purified solution via atomic absorption spectroscopy.

3. Results and Discussion

3.1. Alkaline leaching of CFC

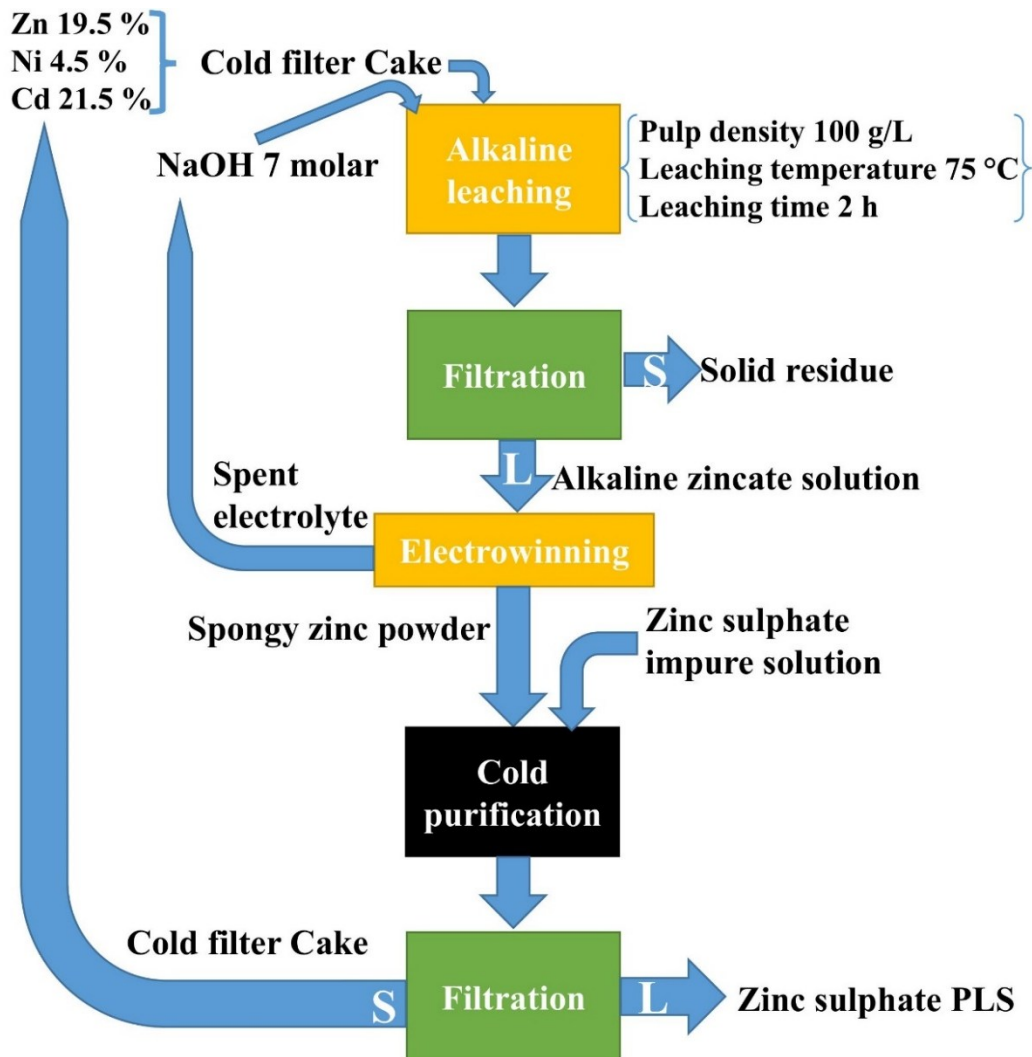
Alkaline leaching offers a selective approach to extracting zinc from complex mixtures such as CFC. Under alkaline conditions, a significant portion (84 %) of zinc is dissolved according to reaction (1) as sodium zincate (Na₂(Zn(OH)₄), while cadmium and nickel remain largely in the solid residue. This selectivity stems from the formation of an anionic zinc complex (ZnO₂²⁻) in the caustic solution. This complex formation effectively solubilizes zinc, allowing for its separation from cadmium and nickel. These latter metals, unlike zinc, exhibit limited solubility in alkaline media, preventing their co-dissolution and

thereby simplifying the subsequent process. Consequently, alkaline leaching represents a promising technique for isolating zinc while minimizing contamination from undesirable metallic impurities.



3.2. Electrowinning to produce zinc powder

The electrowinning of zinc from an alkaline zincate solution, derived from leaching processes, offers a compelling alternative to traditional zinc production methods. This approach leverages direct powder production, bypassing the energy-intensive steps of melting and atomization. In this study, the obtained zincate solution containing 16.5 g/L zinc was utilized as an electrolyte at a current density of 463 A/m². After a 10-hour electrowinning process, the zinc concentration decreased to below 3 g/L.



Figs. 1. Flowchart of experiments.

While Faraday's Laws predicted a zinc deposition of 61 g on the stainless-steel cathode, only 50 g of zinc powder was recovered, resulting in a current efficiency of approximately 82 %. This reduced efficiency

is attributed to the depletion of zincate concentration during the process. The resulting deposit exhibited a spongy, powdery morphology, readily amenable to purification through washing. The product boasts a high purity (>99 %) as evidenced by impurity analysis in [Table 1](#). Despite the less-than-ideal current efficiency, the direct powder production method holds promise for a more sustainable and efficient zinc extraction pathway.

Table 1. Zinc powder's impurity chemical analysis.

Elements	Cd	Ni	Co	Mn	Fe	Cu
Content (mg.kg ⁻¹)	725	53	7	49	112	12

3.3. Cold purification using spongy zinc powder

The efficiency of spongy zinc powder obtained from electrowinning in the cold purification stage of metal extraction processes is significantly affected by its inherent composition and the cementation conditions employed. The initial analysis of the spongy zinc powder, as referenced in [Table 1](#), reveals cadmium as the main impurity. This necessitates careful consideration of process parameters to optimize the removal of cadmium and other impurities like nickel. Due to the carbonation or oxidation of zinc metal when exposed to air, these powders are first washed in a dilute sulfuric acid solution for one minute and then immediately placed in water to be used in the cold purification stage.

Cementation experiments, summarized in [Table 2](#) and visualized in [Fig. 2](#), demonstrate the reduction of nickel and cadmium impurities through metallic zinc precipitation. Initial trials using 4 g of spongy zinc powder over an hour reaction period demonstrably reduced the concentrations of nickel and cadmium to 5.7 and 1.6 mg.L⁻¹, respectively. While this initial success validated the basic principle of cementation, the resulting nickel concentration of 5.7 mg.L⁻¹ remained unacceptably high for direct electrowinning. This finding indicated a need for further optimization of the experimental parameters to achieve the required levels of purity.

Table 2. Conditions and results of cold purification using spongy zinc powder obtained from alkaline electrowinning.

Experiment	Conditions		Impurity concentration (mg. L ⁻¹)	
	Zinc powder amount (g)	Time (h)	Ni	Cd
1	4	1	5.7	1.6
2	4	2	4	2.5
3	5	1	1.5	1.4

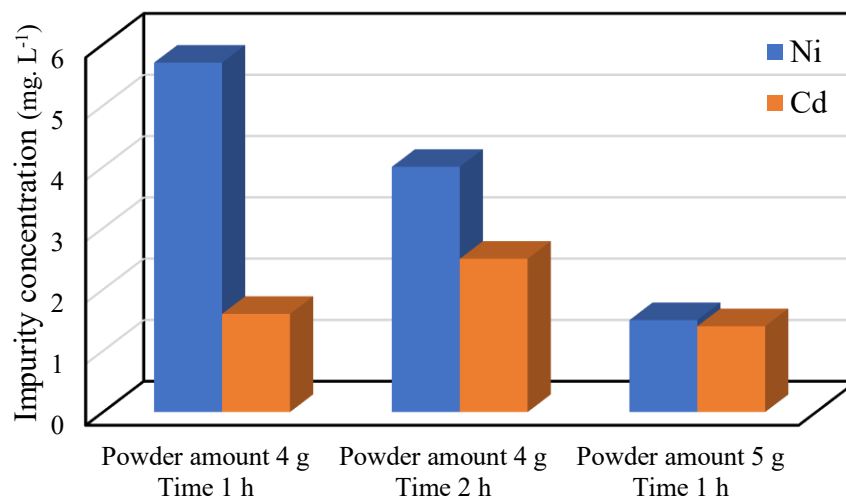


Fig. 2. Impurity concentration after cold purification using spongy zinc powder obtained from alkaline electrowinning.

Extending the purification time to two hours, in an attempt to further reduce the nickel concentration, yielded unexpected and problematic results. While the nickel concentration did decrease, a paradoxical increase in the cadmium concentration was observed, suggesting the re-dissolution of previously cemented cadmium. This phenomenon underscores the importance of understanding the dynamic equilibrium governing the cementation process. Factors such as the limited availability of zinc surface area for continued deposition and alterations in the solution chemistry over time can contribute to the re-dissolution of previously precipitated metals.

The ultimate successful optimization of the cold purification process was achieved by increasing the dosage of spongy zinc powder to 5 g. This single modification led to a significant reduction in both cadmium and nickel concentrations, reaching levels of 1.4 and 1.5 mg.L⁻¹, respectively. These concentrations are deemed acceptable for direct use in the electrowinning process. The success of this adjustment highlights the crucial role of sufficient zinc powder availability in driving the cementation reaction to completion. A greater quantity of zinc provides more surface area for the effective scavenging of both cadmium and nickel impurities, minimizing the potential for re-dissolution and ensuring efficient purification.

4. Conclusions

Alkaline leaching is an effective method for selectively extracting zinc from complex mixtures like CFC (cadmium, nickel, and zinc) containing materials. The process dissolves a large portion (84 %) of zinc as sodium zincate under alkaline conditions, while cadmium and nickel largely remain in the solid residue. This treatment simplifies the separation process, minimizing contamination from undesired metallic impurities. This method offers a promising technique for isolating zinc efficiently while reducing the presence of other unwanted metals. The study focuses on electrowinning zinc from a zincate solution as an environmentally friendly alternative to conventional zinc production methods. Current efficiency was around 82 %, lower due to zincate concentration depletion. But the produced zinc deposit has a spongy, powdery structure that allows easy purification, and zinc powder has high purity (>99 %). Despite the efficiency challenges, the direct powder production method shows promise for sustainable and efficient zinc extraction. The efficiency of spongy zinc powder in cold purification during metal extraction depends

on its composition and the cementation conditions used. The powder, containing cadmium as the main impurity, requires careful optimization to remove cadmium and other impurities like nickel efficiently.

Utilizing zinc powder derived from CFC offers several advantages. Firstly, it minimizes waste disposal and contributes to a more sustainable process. Secondly, it reduces the reliance on purchased zinc dust, leading to significant cost savings. Finally, by controlling the production process, the purity and reactivity of the zinc dust can be tailored to optimize the cold purification stage, ensuring efficient removal of detrimental impurities and ultimately contributing to the production of high-grade zinc metal. Therefore, the recovery of zinc from CFC for use in cold purification represents a valuable and economically viable practice in modern zinc hydrometallurgy.

Ethical Considerations

The authors avoided data fabrication, falsification, and plagiarism, and any form of misconduct.

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Conflict of Interest

The authors declare no conflict of interest.

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