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Response surface methodology optimization-ultrasonic enhanced zinc powder purification of copper and cadmium in zinc sulfate solution

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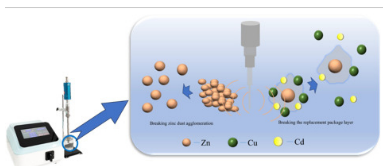
Highlights

- Ultrasonic technology was introduced to enhance the zinc powder displacement process by leveraging its cavitation and mechanical effects. This approach effectively mitigated issues related to zinc powder passivation and agglomeration, thereby overcoming the conventional limitations in purification efficiency.
- Response surface methodology was applied to systematically optimize the ultrasonic purification process, enabling the identification of critical parameter interactions. Under the optimized conditions—specifically, a reaction time of 30 min, ultrasonic power of 120 W, and a zinc powder dosage of 2.6 times the theoretical amount—the removal efficiencies for cadmium and copper reached 48.16 % and 98.10 %, respectively.
- The mechanical effects induced by ultrasound resulted in the refinement of purification residue particles to an average size of 8.16 μm, which substantially enhanced the utilization efficiency of the zinc powder.

Abstract

This research examined the underlying mechanisms and effectiveness of employing ultrasonic intensification in the zinc powder cementation purification process aimed at removing copper and cadmium impurities from zinc sulfate solutions within hydrometallurgical applications. The findings indicate that ultrasonic cavitation efficiently eliminates the inert passivation layer present on the surface of zinc powder, prevents particle agglomeration, and maintains a highly reactive surface area. The micro-jets and shock waves generated by the collapse of cavitation bubbles substantially enhance the mass transfer rates of copper and cadmium ions to the zinc powder surface, thereby accelerating the cementation reaction. Additionally, the thermal and mechanical effects induced by ultrasound contribute to improved uniformity and stability of the reaction system. Process parameters were optimized through Response Surface Methodology employing a Box-Behnken Design, resulting in optimal conditions identified as a reaction time of 30 min, ultrasonic power of 120 W, and a zinc powder dosage at 2.6 times the stoichiometric requirement. Notably, the application of ultrasound significantly reduced the average particle size of the purified residue from 31.89 μm, observed in the conventional process, to 10.88 μm, primarily attributable to ultrasonic grain refinement facilitated by high-frequency mechanical agitation. This pronounced decrease in particle size markedly enhances zinc powder utilization efficiency.

Graphical abstract



Introduction

Zinc, as a fundamental metal material essential to national economic development, finds extensive applications in zinc plating, alloy production, battery manufacturing, the chemical industry, and various other sectors. With the rapid advancement of global industrialization, the demand for zinc has been steadily increasing. Currently, zinc production is primarily categorized into two main processes: pyrometallurgical zinc smelting and hydrometallurgical zinc smelting [10,16,18,19]. Zinc hydrometallurgy has emerged as the predominant method for zinc production globally, owing to its advantages of low energy consumption, environmental sustainability, and high metal recovery efficiency. This process currently accounts for over 85 % of the total zinc production worldwide [2,17]. The traditional wet zinc refining process encompasses several critical stages, including the roasting of zinc concentrate, sulfuric acid leaching, purification and removal of impurities from the leachate, and the electro-winning of zinc sulfate solution. Following the roasting step, the zinc concentrate is converted into calcine, which is subsequently leached with sulfuric acid to produce a zinc sulfate solution. Nevertheless, this solution generally contains a range of impurity elements, such as copper, cadmium, cobalt, and nickel. The presence of these impurities, if not adequately eliminated, can lead to significant adverse effects during the subsequent electro-winning phase [9].

In the hydrometallurgical refining of zinc, the efficient purification of zinc sulfate leach solutions constitutes a critical stage to guarantee the stable and effective performance of the subsequent electrolytic deposition process, thereby enabling the production of high-purity cathode zinc [13,20]. However, the industrial zinc sulfate leaching solution often contains a variety of impurity metal ions, among which copper (Cu) and cadmium (Cd) are particularly harmful to the zinc electrowinning process because of their similar electrode potential to zinc [23]. The presence of these impurities will not only reduce the grade of cathode zinc, increase energy consumption, and even lead to the " burning plate " phenomenon in the electrolysis process, affecting the continuity and economy of production [14,21,24].

At present, zinc powder replacement method is widely used in industry to remove copper, cadmium and other impurities in zinc sulfate solution. This method utilizes the reducibility of zinc to reduce metal ions such as copper and cadmium in the solution to metal elements and precipitate separation. Although the zinc powder replacement method is theoretically feasible and widely used, the process still faces many challenges in actual industrial production. Firstly, the hydrolysis and passivation of zinc powder are the key factors restricting its utilization efficiency [11]. The surface of newly added zinc powder is easy to react with water or dissolved oxygen in the solution to form hydroxide or oxide film, which hinders the effective contact between zinc powder and impurity ions in the solution, thus reducing the activity of zinc powder and increasing the actual consumption of zinc powder, which is much higher than the theoretical calculation value. Secondly, the agglomeration of zinc powder particles further deteriorates the purification effect [10,15]. The fine zinc powder particles are easy to agglomerate due to the high surface energy, forming a large particle group, reducing the total specific surface area of the zinc powder, reducing the effective reaction area of the zinc powder and slowing down the reaction rate. In addition, the traditional zinc powder replacement process usually needs to be carried out at a higher temperature, which not only increases energy consumption, but also may aggravate the hydrolysis and oxidation of zinc powder [29]. More importantly, the addition of excessive zinc powder and the failure to make full use of zinc powder will eventually enter the purification residue, which not only causes the waste of zinc resources, but also may bring the risk of secondary pollution due to the high content of heavy metals in the purification residue, which increases the difficulty and cost of subsequent purification residue treatment. Therefore, it is of great practical significance and economic benefits to develop a new purification technology that can effectively overcome the defects of traditional zinc powder replacement process, improve the utilization rate of zinc powder, and reduce the risk of energy consumption and secondary pollution [5,7].

Concerning the displacement residue produced during the displacement process, two primary technical approaches are currently employed: pyrometallurgical and hydrometallurgical methods. The pyrometallurgical approach, having been implemented earlier, is technologically well-established and demonstrates superior metal recovery efficiency. Nevertheless, this method is characterized by high energy consumption, dependence on costly smelting coke, and necessitates the installation of systems for furnace ash recovery and exhaust gas purification, all of which contribute substantially to environmental burdens.

In contrast, Liu et al [7]. developed a hydrometallurgical copper enrichment technology based on waste electrolyte combined with online pH monitoring, used for selective leaching of zinc, cadmium, and cobalt from copper-cadmium residue. This study optimized process parameters using the Box-Behnken design (RSM-BBD) in response surface methodology, determining the optimal conditions as: sulfuric acid concentration 125 g/L, reaction temperature 42.5 °C, reaction time 240 min, and liquid-to-solid ratio 4.5:1. Under these conditions, the leaching efficiencies of zinc, cadmium, and cobalt reached 96.84 %, 96.12 %, and 96.37 %, respectively, with a residue rate of 9.1 % after reaction. The copper content in the leached residue was significantly increased to 56.8 %, achieving effective copper enrichment. Although the copper loss rate during the leaching stage was 18.34 %, after implementing two-stage copper recovery through online pH regulation, the copper concentration in the solution was reduced to <0.1 mg/L, thereby greatly reducing copper loss while efficiently recovering copper.

This process innovatively utilizes waste electrolyte to treat copper-cadmium residue, not only alleviating the treatment pressure of waste electrolyte but also enhancing the resource value of copper-cadmium residue, providing a feasible path for the synergistic treatment of industrial waste residue and waste liquid.

The integration of ultrasound with hydrometallurgy is considered a green process primarily due to its physical intensification of reaction mechanisms, which systematically adheres to the principles of green chemistry. Ultrasonic technology, recognized as an effective and environmentally friendly physical enhancement technique, has demonstrated extensive potential for application within the domain of hydrometallurgy [12]. The fundamental mechanism is based on the cavitation phenomenon induced by the transmission of ultrasonic waves through liquid media [7].

When the energy of the ultrasonic wave reaches a sufficiently high level, the microscopic bubbles within the liquid experience a sequence of formation, growth, contraction, and violent collapse. This phenomenon is associated with extreme physical and chemical conditions, including localized elevated temperatures, high pressures, micro-jet formation, and shock waves [6]. These phenomena can substantially influence mass transfer, the reaction interface, and the properties of particles within metallurgical processes. In particular, the micro-jets produced by ultrasonic cavitation exert a pronounced effect on the surfaces of solid particles by removing passivation films or adsorbed layers, thereby exposing fresh, reactive surfaces and enhancing the reaction rate. Concurrently, the micro-jets and associated shock waves facilitate particle dispersion, inhibit agglomeration, and consequently increase the specific surface area available for reaction [10]. Furthermore, the transient high-temperature and high-pressure conditions produced during the collapse of cavitation bubbles can alter the activation energy of chemical reactions, facilitate the cleavage of certain chemical bonds, and promote the formation of new bonds, thereby accelerating the overall reaction kinetics. In the field of hydrometallurgy, ultrasonic technology has been effectively employed in various processes, including mineral leaching, solvent extraction, metal electrodeposition, and wastewater treatment [25,27,28].

The principal advancement presented in this research lies in the integration of the physical effects of ultrasound—particularly the cavitation phenomenon—with the conventional zinc powder chemical replacement purification method, thereby synergistically enhancing purification efficiency and addressing the limitations inherent in existing technologies. In traditional zinc powder purification, issues such as particle agglomeration and surface passivation (e.g., the formation of hydroxide coatings) reduce the effective reactive surface area and diminish zinc powder activity. These factors contribute to excessive zinc consumption, prolonged purification durations, and suboptimal impurity removal performance. The application of ultrasonic waves, especially through cavitation effects, generates transient localized high temperatures, elevated pressures, intense microjets, and shock waves within the liquid medium. Such phenomena effectively remove passivation layers from zinc powder surfaces, inhibit particle agglomeration, and sustain high reactivity levels. This study systematically investigated critical process parameters—including ultrasonic power, reaction time, solution temperature, and zinc powder dosage—employing response surface methodology to analyze experimental data. The findings elucidate the individual and interactive effects of these variables on the removal efficiencies of copper and cadmium, thereby advancing the mechanistic understanding of the ultrasound-enhanced zinc powder replacement reaction. Consequently, this work establishes a novel, efficient, stable, and cost-effective purification process for copper and cadmium, underpinned by ultrasonic enhancement of zinc powder reactivity. The developed methodology offers significant technical support for the environmentally sustainable and green advancement of the zinc hydrometallurgy industry.

Section snippets

Experimental methods

The response surface method (RSM) employs mathematical and statistical approaches to develop experimental models for optimizing responses (output variables) influenced by multiple independent variables [4,8]. Using Box-Behnken design (BBD), this study analyzed the effects of time (X_1), ultrasonic power (X_2), and amount of zinc powder (X_3) on copper (Y_1) and cadmium (Y_2) removal rates [30]. Optimization was performed with Design-Expert 10.0 software to determine optimal factor levels and …

Experimental results

As depicted in Fig. 2(a), when the quantity of zinc powder ranges from 1.8 to 3.4 times, the cadmium removal rate exhibits a gradual decline with prolonged reaction time. Nevertheless, at a constant reaction duration, increasing the zinc powder amount from 1.8 to 3.4 times results in a marked enhancement of the cadmium removal rate, rising from 22.43 % to 69.21 %. This phenomenon can be attributed to the greater availability of reactants for the displacement reaction afforded by the increased …

Conclusion

(1) The optimal processing parameters identified through the Response Surface Methodology-Box-Behnken Design (RSM-BBD) model were a reaction time of 30 min, ultrasonic power set at 120 W, and a zinc powder dosage of 2.6 times. Under these conditions, the cadmium removal efficiency reached 48.16 %, while the copper removal efficiency attained 98.10 %. …

(2) Application of ultrasonic treatment resulted in a reduction of the average particle size from 31.89 μm in conventional purification residues to 10.88 …

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CRediT authorship contribution statement

Shixiong Peng: Writing – original draft, Validation, Supervision, Software, Resources, Methodology, Conceptualization. **Jianwei Zhu:** Supervision, Resources, Methodology. **Yingjie Xu:** Writing – original draft, Methodology, Data curation. **Hongwei Li:** Supervision, Methodology. **Hongying Xia:** Methodology, Funding acquisition, Data curation. **Yongliang Zhu:** Supervision, Software. **Wentao Zheng:** Software, Resources. **Haiyang Liu:** Supervision, Software. **Libo Zhang:** Writing – original draft, Methodology, …

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. …

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