

The Effect of H_2SO_4 Concentration on the Yield and Purity of Zinc Sulfate in the Synthesis of Zinc Sulfate from Zinc Waste

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Abstract

Zinc waste is a byproduct of industrial activities originating from the production and utilization of zinc metal, particularly in manufacturing sectors such as the paint, ceramics, glass, lighting equipment, and pesticide industries. Without proper management, this waste has the potential to cause significant environmental impacts, especially through water pollution. Zinc plays an essential role as a trace element in aquatic ecosystems, but at levels exceeding the threshold, it can be toxic to aquatic organisms and soil biota. Overexposure to zinc can disrupt the ecological balance, reduce environmental quality, and increase the risk of health problems in humans, including digestive disorders and damage to certain organs. This study aims to analyze the effect of variations in sulfuric acid (H_2SO_4) concentration on the yield and purity of zinc sulfate synthesized from zinc waste. The synthesis procedure included a pre-treatment stage consisting of particle size reduction, immersion in hydrochloric acid (HCl) to remove the oxide layer, surface cleaning, and impurity separation. The reaction was carried out using H_2SO_4 with concentrations of 20%, 30%, 35%, and 45% at a temperature of 75–80 °C with a stirring speed of 300–350 rpm, followed by filtration, concentration, and crystallization. Optimal conditions were achieved at a 45% H_2SO_4 concentration, which produced white heptahydrate zinc sulfate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) crystals with a mass of 3.1729 g, a content of 52.80%, and a purity of 96.28%.

Keywords: *zinc waste, sulfuric acid, zinc sulfate, yield, complexometric*

Abstrak

Limbah seng merupakan hasil samping aktivitas industri yang berasal dari proses produksi dan pemanfaatan logam seng, khususnya pada sektor manufaktur seperti industri cat, keramik, kaca, peralatan penerangan, dan pestisida. Tanpa pengelolaan yang memadai, limbah ini berpotensi menimbulkan dampak lingkungan yang signifikan, terutama melalui pencemaran perairan. Seng berperan sebagai unsur esensial dalam konsentrasi jejak bagi ekosistem akuatik, namun pada kadar yang melebihi ambang batas dapat bersifat toksik bagi organisme perairan dan biota tanah. Paparan seng berlebih dapat mengganggu keseimbangan ekologi, menurunkan mutu lingkungan, serta meningkatkan risiko gangguan kesehatan pada manusia, termasuk gangguan pencernaan dan kerusakan organ tertentu. Penelitian ini bertujuan menganalisis pengaruh variasi konsentrasi asam sulfat (H_2SO_4) terhadap rendemen dan kemurnian seng sulfat yang disintesis dari limbah seng. Prosedur sintesis meliputi tahap pra-perlakuan berupa pengecilan ukuran partikel, perendaman dalam asam klorida (HCl) untuk menghilangkan lapisan oksida, pembersihan permukaan, dan pemisahan pengotor. Reaksi dilakukan menggunakan H_2SO_4 berkonsentrasi 20%, 30%, 35%, dan 45% pada suhu 75–80 °C dengan kecepatan pengadukan 300–350 rpm, dilanjutkan filtrasi, pemekatan, dan kristalisasi. Kondisi optimum dicapai pada konsentrasi H_2SO_4 45% yang menghasilkan kristal putih seng sulfat heptahidrat ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) dengan massa 3,1729 g, kadar 52,80%, dan kemurnian 96,28%.

Kata kunci: *limbah seng, asam sulfat, seng sulfat, rendemen, kompleksometri*

1. Introduction

The metalworking industry produces various types of solid waste, one of which is zinc powder and scrap formed from the processes of lathe cutting, cutting, and metal surface smoothing.[1], [2]. This zinc waste is generally not managed optimally because it is considered non-hazardous and of low economic value, so it is often simply discarded or stockpiled without further treatment.[3], [4], [5]. In fact, zinc is a metal that has strategic value and plays an important role in various applications, such as catalysts.[6], [7], galvanization materials[8], [9], [10], [11], batteries[12], [13], micro fertilizers[14], [15], [16], [17], and pharmaceutical raw materials[18], [19], [20], [21].

Zinc sulfate (ZnSO_4) is one of the zinc-based compounds widely used in the fertilizer[22], [23], [24], [25], textile[26], [27], [28], electrochemistry[29], [30], [31], [32], pharmaceutical[33], [34], [35], and metal coating industries[36], [37], [38]. The demand for ZnSO_4 continues to increase, while conventional production generally relies on primary zinc ores such as sphalerite, which are increasingly limited in availability and require energy-intensive and costly extraction processes[8][39]. Therefore, converting zinc waste into ZnSO_4 through a hydrometallurgical approach is a relevant strategy to promote waste utilization in support of the circular economy and green chemistry[40], [41], [42], [43], [44].

Synthesis of zinc waste into zinc sulfate using a synthesis process with sulfuric acid at several different concentrations. In another study, 16% sulfuric acid and hydrogen peroxide were used with a reaction time of 20 minutes.[45]. In another study, Zn-C battery waste was converted into zinc sulfate compounds using sulfuric acid with concentrations of (5%, 10%, 15%, 20%, 25%, and 30%) in a volume of 80 ml, and the maximum result was obtained with 30% sulfuric acid, producing approximately 18.39 grams of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ crystals with an efficiency value of 83.71%[46].

Hydrometallurgical processes such as leaching using sulfuric acid, followed by purification, filtration, and crystallization, have the potential to produce high-purity ZnSO_4 if the process conditions are optimized[47], [48]. In addition to providing added value, this approach can also reduce the burden of environmental pollution caused by unmanaged metal waste[49], [50], [51], [52], [53]. However, to date, research related to ZnSO_4 synthesis based on lathe zinc waste is still limited, both in terms of reaction condition optimization, leaching kinetics studies, and product characterization analysis using modern instrumental techniques[43], [54].

The objectives of this study are to determine whether zinc waste can be synthesized into zinc sulfate and to determine the effect of sulfuric acid concentration on the yield and purity of zinc sulfate produced from zinc waste.

2. Materials and Methods

Research Materials and Equipment

The materials used in this study were zinc waste collected from workshops and ongoing projects as the main raw material. Supporting materials used were pro-analytical grade sulfuric acid grade hydrochloric acid (HCl), and distilled water. The tools used are laboratory glassware, analytical balances, hot plates, magnetic stirrers, wire mesh, clamps, and other supporting laboratory equipment.

Preparation and Pre-treatment of Raw Materials

The raw materials to be used need to be treated to remove impurities. The raw material, zinc waste, is washed with soap to remove oil and other impurities, then dried. The dried zinc is sanded first to remove impurities, then cut into small pieces. The zinc pieces are then soaked in a 1N HCl solution for 30 minutes, cleaned, and dried[46].

Zinc Sulfate Synthesis

The next process is the synthesis process. This involves placing the zinc pieces into a sulfuric acid solution with a ratio of 1:8, with a slight excess of sulfuric acid. The solution is heated to 75-80°C and stirred at 300-350 rpm. During the process, a reaction occurs, indicated by the presence of hydrogen gas bubbles because zinc reacts with sulfuric acid, producing a pungent odor. Visually, a cloudy gray solution forms, and over time, the cloudy color becomes clear when left to stand because two solutions will form: the bottom layer is gray, while the top layer is clear[46]. The clear solution in the Erlenmeyer flask, which still contains impurities, is filtered to separate the impurities and the filtrate. The resulting filtrate is then heated to a temperature of 80–90°C until the solution is saturated. The solution is cooled to room temperature, forming crystals that settle at the bottom. The formed crystals are separated by filtration and are white in color. The crystallization process is carried out at room temperature (25–30°C), which is the optimal temperature for crystal formation[45]

Analysis of Zinc Sulfate Yield

The synthesized sample is placed in a crucible. It is then heated at 60°C until dry. It is then weighed using an analytical balance. Zinc sulfate yield analysis uses the weight of dry zinc sulfate. To determine the zinc sulfate yield, it can be calculated according to equations (1) and (2):

$$\text{Weight} = \text{Weight}_{(\text{wet})} - \text{Weight}_{(\text{dry})} \quad (1)$$

$$\text{Rendemen} = \frac{\text{substance weight}}{\text{mixed weight}} \times 100\% \quad (2)$$

Analysis of Zinc Sulfate Purity

The zinc sulfate crystals produced were titrated using complexometric to calculate the zinc sulfate content. To titrate zinc sulfate, a primary solution, a secondary solution, and an indicator solution are required. The primary solution used is the zinc sulfate solution produced, the secondary solution used is 0.05 M Na₂EDTA solution, ammonia buffer solution pH 10, and the indicator used is EBT indicator. Weigh 200 mg of zinc sulfate and dissolve it in 50 ml of distilled water, shaking for 3 minutes. Then add 5 ml of pH 10 ammonia buffer solution and 2 drops of EBT indicator, and the solution will turn grape red. Perform titration with 0.05 M Na₂EDTA until a clear blue color forms (Alfiyanto Rahman, n.d.). To determine the purity level of zinc sulfate, calculate according to equations (3) and (4):

Calculation of ZnSO₄ Weight

$$\text{ZnSO}_4 \text{ weight} = \frac{BM \text{ ZnSO}_4}{BM \text{ ZnSO}_4 \cdot 7H_2O} \times \text{weight} \quad (3)$$

$$\text{ZnSO}_4 \text{ content} = \frac{(V \times M) \text{ Na}_2\text{EDTA} \times BM \text{ ZnSO}_4}{mg \text{ ZnSO}_4} \times 100\% \quad (4)$$

Description:

V = Na₂EDTA titration volume

M = Molarity of Na₂EDTA

3. Results and Discussion

Synthesis of Zinc Sulfate

The results of zinc sulfate synthesis from zinc waste with sulfuric acid are shown in **Figure 1** and **Table 1**.



Figure 1. Zinc waste, zinc waste washing with hydrochloric acid, synthesis of zinc sulfate, and zinc sulfate crystals

Table 1. Results of ZnSO₄ synthesis from zinc waste and sulfuric acid of various concentrations

Sulfuric acid concentration (%)	Crystal Color	ZnSO ₄ yield (%)			Purity Level of ZnSO ₄ (%)		
		1	2	Average	1	2	Average
20	Greenish white	57.31	56.54	56.93	62.35	59.18	60.77
30	Greenish white	85.05	86.61	85.83	71.64	76.74	74.19
35	Greenish white	88.30	87.30	97.80	78.62	85.06	81.84

Sulfuric acid concentration (%)	Crystal Color	ZnSO ₄ yield (%)			Purity Level of ZnSO ₄ (%)		
		1	2	Average	1	2	Average
45	Greenish white	53.11	52.46	52.80	96.90	95.66	96.28

The synthesis of zinc sulfate from the lathe workshop zinc waste through reaction with 20–45% concentrated sulfuric acid solution shows that variations in acid concentration significantly affect the dissolution rate of zinc metal, conversion efficiency, and crystal characteristics of the product[55], [56]. In general, the reaction between zinc and sulfuric acid follows a simple electrochemical mechanism involving the anodic dissolution of zinc ($\text{Zn} \rightarrow \text{Zn}^{2+} + 2\text{e}^-$) and the reduction of protons to hydrogen gas ($2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$)[57], [58]. Increasing acid concentration causes an increase in H-ion activity in the solution, so that the kinetics of the zinc dissolution reaction tend to grow. This is reflected in the shorter reaction time at an acid concentration of 40–45% compared to 20–25%[28], [59], [60], [61].

However, increasing the acid concentration does not always result in purer zinc sulfate crystals. At concentrations above 35%, the dissolution rate is too fast, resulting in high local supersaturation and causing rapid nucleation, which produces relatively smaller crystals. Meanwhile, at low to medium concentrations (20–30%), the nucleation and crystal growth processes are more controlled, resulting in more uniform crystal sizes. However, zinc sulfate crystals with a greenish-white color were obtained at all variations in acid concentration. This phenomenon can be attributed to two possibilities: (1) the presence of transition metal impurities such as Fe^{2+} or Cu^{2+} originating from zinc workshop waste, or (2) the influence of reaction conditions that trigger the formation of temporary complexes that remain in the crystal structure. The light green color indicates that the concentration of impurities is relatively low but still sufficient to affect the visual appearance of the product. [62], [63], [64], [65].

Based on visual analysis and the physical characteristics of the crystals, it can be concluded that all acid concentration variations are capable of converting zinc waste into soluble zinc sulfate, which then crystallizes in the form of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$. [66], [67]. The literature states that hydrated zinc sulfate crystals (monohydrate and heptahydrate) are naturally white in color [68], [69], so the presence of a greenish color is an initial indicator that further purification processes—such as recrystallization or selective adsorption—are still needed if the product is to be used for certain industrial applications[70]. Nevertheless, from the perspective of waste management and chemical synthesis based on the circular economy, this process has shown great potential in converting solid waste into high-yield, value-added chemical products.

Overall, the variation in sulfuric acid concentration of 20–45% provides insight into the relationship between reaction conditions, dissolution mechanisms, and the resulting crystal characteristics. These results reinforce the idea that zinc waste from machine shops can be used as an alternative precursor in the synthesis of zinc sulfate, which is more economical and supports the reduction of metal waste in the environment [71], [72], [73], [74].

The effect of sulfuric acid concentration on zinc sulfate yield

The effect of sulfuric acid concentration on zinc sulfate yield is shown in **Table 1** and **Figure 2**.

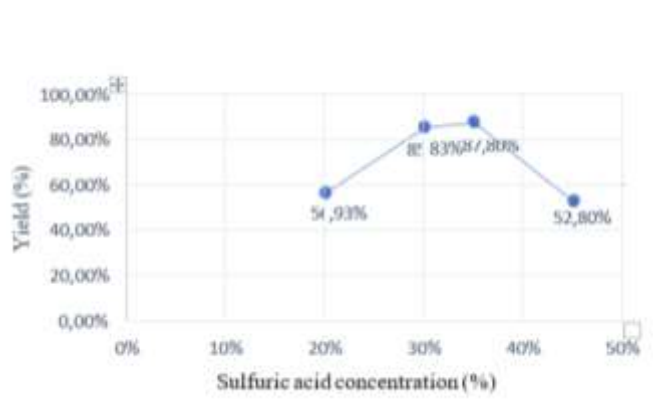


Figure 2. The relationship between sulfuric acid concentration and zinc sulfate yield

The results of the study indicate that sulfuric acid concentration has a very significant effect on the yield of zinc sulfate synthesized from lathe workshop zinc waste. The pattern of yield change is not linear,

but shows an optimum reaction point at a certain concentration. The yield increases sharply from 56.93% at a concentration of 20% to 85.83% at 30%, then reaches its highest value of 97.80% at a concentration of 35%. However, at a concentration of 45%, the yield actually decreased dramatically to 52.80%. This phenomenon indicates that the efficiency of zinc conversion is influenced by the equilibrium between the metal dissolution rate and the supersaturation conditions of the solution during the synthesis process[75], [76], [77], [78], [79].

The increase in yield from a concentration of 20% to 35% shows that the higher the acid concentration, the higher the activity of H^+ ions in the solution, thereby increasing the rate of zinc dissolution ($Zn + H_2SO_4 \rightarrow Zn^{2+} + SO_4^{2-} + H_2\uparrow$)[79], [80], [81]. In the 20–35% concentration range, the solution conditions are still stable enough so that Zn^{2+} and SO_4^{2-} ions can react to form $ZnSO_4$ complexes optimally without producing kinetic disturbances such as evaporation, thermal degradation, or the formation of a passivation layer. A concentration of 35% is the optimum point because under these conditions, the rate of Zn^{2+} formation is high but still balanced with the diffusion and mixing mechanisms of the solution, so that almost all of the zinc is converted to zinc sulfate. This is in line with the theory that the dissolution reaction of metals in mineral acids has an optimum zone when reactivity is high but does not yet cause extreme local supersaturation conditions[82], [83].

The decrease in yield at a concentration of 45% to 52.80% indicates the negative effect of excessively high acid concentration. At this concentration, the reaction proceeds so rapidly that large amounts of hydrogen gas are formed, which can inhibit the uniform dissolution of zinc by forming gas bubbles that adhere to the metal surface. In addition, the high concentration of H_2SO_4 can trigger the formation of a passivated layer due to surface oxidation or the presence of impurities from workshop zinc waste, so that some of the zinc does not react completely. Reactions that are too fast can cause an imbalance between the rate of zinc dissolution and the rate of $ZnSO_4$ complex formation, so that some Zn^{2+} ions are not converted into soluble products and may even be precipitated back as oxides/hydroxides if local conditions change due to an increase in reaction temperature[3], [4], [7], [11], [82], [83].

In addition to reaction kinetics, this phenomenon can also be explained through crystallization control[84], [85], [86], [87]. At 20–35%, the concentration of Zn^{2+} and SO_4^{2-} ions in the solution is within a range that supports the stable formation of $ZnSO_4 \cdot 7H_2O$ crystals. At 45%, very rapid supersaturation can result in uncontrolled nucleation, so that most of the product does not crystallize completely or is in an amorphous form that is not measured as crystal yield. This condition is common in reaction systems with very high ion gradients[88], [89].

Overall, the results of this study confirm that sulfuric acid concentration is a key parameter in the synthesis of zinc sulfate from zinc waste[32], [90], [91], [92]. A concentration of 35% proved to be the optimum point for obtaining high yields, while low concentrations (<30%) did not provide sufficient protons for metal dissolution, and excessively high concentrations (>40%) caused kinetic and thermodynamic imbalances that reduced yields. These findings are crucial for optimizing zinc-based waste production processes at both laboratory and industrial scales, while also reinforcing the potential for utilizing metal waste as a valuable chemical feedstock within the circular economy concept[93], [94], [95], [96].

The effect of sulfuric acid concentration on the purity level of zinc sulfate

The effect of sulfuric acid concentration on zinc sulfate yield is shown in **Table 1** and **Figure 3**.

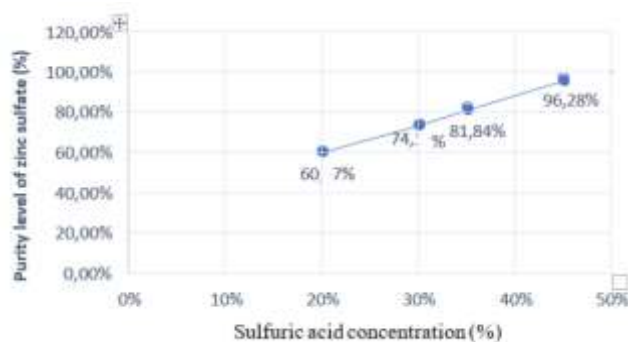


Figure 3. The relationship between sulfuric acid concentration and zinc sulfate purity

The effect of sulfuric acid concentration on the purity level of zinc sulfate shows a different pattern compared to its effect on yield. The data shows that an increase in sulfuric acid concentration from 20% to 45% results in a relatively consistent upward trend in purity, from 60.77% (20%) to 96.28% (45%). This indicates that higher acid concentrations not only accelerate zinc dissolution but also enhance the separation process from other metal impurities contained in the lathe workshop zinc waste[90], [91], [97], [98], [99].

At sulfuric acid concentrations of 20–30%, the purity of zinc sulfate is still at a moderate level (60.77–74.19%). The low purity in this range is likely influenced by two main factors: first, the H^+ ion activity is not yet optimal, so that the dissolution of zinc metal is not yet complete. Under these conditions, some metal impurities, such as Fe, Cu, or Al from zinc waste, are still dissolved or trapped in the solution phase, making them difficult to separate at the crystallization stage[98], [100], [101], [102]. Second, the relatively low acid concentration causes the formation rate of Zn^{2+} and SO_4^{2-} ions to be not very high, resulting in slow crystallization and allowing impurity particles to be incorporated into the $ZnSO_4 \cdot 7H_2O$ crystal matrix.

Increasing the sulfuric acid concentration to 35% resulted in an increase in purity to 81.84%. At this concentration, zinc dissolution occurs more completely, resulting in the formation of large amounts of stable Zn^{2+} ions. At the same time, some impurity metals that are more resistant to dissolution in medium sulfuric acid—particularly small amounts of Fe^{2+} and Al^{3+} —tend to precipitate again or are no longer carried in dissolved form. This creates more selective conditions in the zinc sulfate crystal formation process, resulting in a significant increase in purity compared to acid concentrations of 20–30%.

An interesting phenomenon occurs at a concentration of 45%, where the purity of zinc sulfate reaches its highest value, namely 96.28%. These results can be explained through several scientific mechanisms. First, at very high acid concentrations, zinc dissolution occurs very rapidly and produces Zn^{2+} ions with a dominant abundance compared to impurity ions[103], [104], [105], [106]. The ratio of Zn^{2+} to impurity ions increases dramatically, causing the formed crystal phase to be dominated by zinc sulfate with very few contaminants. Second, some metal ions that are unstable in strong acid conditions, such as Fe^{2+} and Cu^{2+} , can undergo hydrolysis or form complexes that do not co-crystallize with $ZnSO_4 \cdot 7H_2O$ compounds, thereby increasing product purity. Third, supersaturation conditions at high acid concentrations accelerate the nucleation of zinc sulfate so that impurities do not have enough time to be trapped in the crystal structure.

This pattern of increased purity differs from the previous synthetic yield trend, which reached an optimum point at 35%. This shows that although the reaction at 45% produces a lower yield, the crystal purification process at high concentrations is actually more efficient. In other words, the high purity at 45% is likely influenced by selective reaction conditions, not by the high conversion rate of zinc to zinc sulfate. The scientific implication is that the optimum conditions for yield and purity are not always at the same concentration and need to be optimized based on the end goal of the product.

Overall, increasing the sulfuric acid concentration has a positive effect on product purity, mainly due to the increased selectivity of zinc dissolution compared to impurities and the limited co-precipitation of impurities under supersaturated conditions[107], [108], [109], [110]. These findings are important for designing zinc waste-based synthesis strategies, both for laboratory needs and larger-scale production, as they show that acid concentration selection can be directed based on specific needs—maximum yield or maximum purity.

4. Conclusion

The conclusions of the study are: 1) Zinc waste can be synthesized into zinc sulfate using sulfuric acid solution to obtain white crystals. 2) Increasing the concentration of sulfuric acid results in a significant yield. After conducting the research, the optimal sulfuric acid concentration used was 45%, which produced a zinc sulfate yield of 52.80% and a zinc sulfate purity level of 96.28%. These findings are important for designing zinc waste-based synthesis strategies, both for laboratory needs and larger-scale production, as they show that acid concentration selection can be directed based on specific needs—maximum yield or maximum purity.

5. References

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