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Removal of Tin and Copper from Semiconductor Wastewater via Hydroxide Precipitation

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ABSTRACT

The rapid growth of the semiconductor industry has led to increased water consumption in production processes, resulting in wastewater containing heavy metals such as tin (Sn) and copper (Cu), which must be treated before being discharged into the environment. This study aims to evaluate the effectiveness of chemical precipitation using sodium hydroxide (NaOH) in recovering Sn and Cu from the semiconductor wastewater. The NaOH concentrations of 1.0 M, 2.5 M, and 4.0 M were added to the wastewater and allowed to undergo mixing using flocculator. The resulting precipitate was analysed through Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Field emission scanning electron microscopy with energy-dispersive X-ray (FESEM-EDX) spectroscopy to determine the functional groups, chemical composition and structure of the precipitate, respectively. The results indicated that 4.0 M NaOH was the optimum concentration for metal precipitation within 30 minutes, achieving 86.8% removal of Sn and 46.2% removal of Cu. FTIR analysis revealed that the precipitate contained metal–O_2 stretching vibrations, while XRD confirmed the presence of tin hydroxide ($Sn(OH)_2$). The precipitate exhibited a rough and textured morphology, characterized by small particles forming aggregates and fine fibrous networks. The precipitation method is a viable option for rapidly treating semiconductor wastewater to meet allowable discharge limits, thereby protecting the environment while enabling the industry to grow without causing significant ecological impacts.

Keywords: Heavy metals; tin; semiconductor wastewater; chemical precipitation

INTRODUCTION

The rapid global expansion of the semiconductor industry has resulted in a substantial rise in water consumption. The industry requires ultra-pure water to clean silicon chips (Noor et al. 2020). Consequently, this has led to the generation of wastewater containing hazardous heavy metals, such as tin (Sn), copper (Cu), lead (Pb), nickel (Ni), aluminium (Al), iron (Fe) and zink (Zn) (Tran et al. 2026). Unproper or incomplete treatment, it can contaminate natural water sources, affect aquatic ecosystems and potentially pose risks to human health (Davis 2022; Teow et al. 2022).

Treatment of semiconductor wastewater is crucial to reduce the risks associated with these heavy metals. The wastewater can be treated through physical, chemical or biological treatment (Sim et al. 2023). Chemical precipitation is a well-established used to remove metal contaminants from the wastewater, particularly in industrial settings where large volumes of water involved (Fu & Wang, 2011). While biological methods can be effective for organic pollutants, they are often inadequate for treating semiconductor wastewater with high concentrations of heavy metals, which require more specialized treatment processes (Qasem et al. 2021).

Chemical precipitation of heavy metals works by adding precipitating agents such as hydroxide, sulphide,

and carbonate into the wastewater (Estay et al. 2021; Lee et al. 2024; Pohl 2020). NaOH reacts with dissolved metal ions to form insoluble metal hydroxides, which can be separated through filtration or sedimentation (Chiu et al. 2019). This process is highly effective for removing metals like tin and copper, which are commonly found in semiconductor wastewater. The use of NaOH is advantageous due to its low cost, ease of use and high efficiency in treating wastewater with high concentrations of heavy metals (Oladimeji et al. 2024; Vlasopoulos et al. 2023). The pH level of the wastewater and the concentration of NaOH are crucial factors that influence the precipitation process, with optimal conditions is required to maximize the removal of contaminants (Wang et al. 2018).

The objective of this study was to evaluate the effectiveness of NaOH in removing tin and copper from the semiconductor wastewater. Two operational factors i.e., initial NaOH concentration and precipitation times were assessed to ensure the treated wastewater meets environmental standards before being discharged to the water body. First and second-order kinetic models were used in this study to understand, quantify and predict the pollutants removal over the times. The findings of this study will contribute to enhancing wastewater treatment practices in the semiconductor industry. In addition, the precipitate was characterized through morphological, elemental, and crystallization analysis.

MATERIALS AND METHODS

SOURCE OF SEMICONDUCTOR WASTEWATER

The semiconductor wastewater was collected from a semiconductor manufacturing plant located in Seremban, Negeri Sembilan, Malaysia. The wastewater was acidic with a pH of 3.10. The heavy metals in the wastewater such as Sn and Cu were 5.57 and 0.45 mg/L, respectively. In addition, the level of COD, ammoniacal-nitrogen, and total suspended solid (TSS) were 349, 7.25, and 65 mg/L, respectively.

SETUP OF PRECIPITATION EXPERIMENTS

The wastewater was pre-treated using a vacuum filtration to remove the TSS. After pre-treatment, about 500 mL of the filtered wastewater was placed into a beaker. NaOH (R&M Chemicals, Malaysia) was added to the beaker at varying concentrations of 1.0, 2.5, 4.0 M. A set of control samples was also prepared, where no NaOH was added to serve as a baseline for comparison. The mixture was stirred using flocculator and allowed to react for three different time intervals i.e., 10, 20 and 30 minutes, while the pH was continuously monitored during the process. The flocculator was operated at a high speed of 100 rpm for the first 3 minutes to initiate the precipitation process and then reduced to a low speed of 30 rpm to allow flocculation occurred. About 40 mL of samples were taken every 5 minutes to study the kinetics of the reaction. The samples were analyzed to observe changes in the concentrations of Sn and Cu over the time. To ensure the reliability and reproducibility of the results, the experiment was repeated three times for each concentration of NaOH.

ANALYTICAL METHODS

The precipitate and treated wastewater was separated by vacuum filtration. The quality of the treated wastewater was analyzed by measuring various water quality parameters, including pH, Sn, Cu, COD, ammoniacal-nitrogen, dissolved oxygen (DO), TSS and colour. pH was measured using a pH meter (Model HQ4300, HACH, United States), while DO was measured with a DO meter (Model HQ1130, HACH, United States). The Sn and Cu were analyzed using inductively coupled plasma mass spectrometry (ICP-MS). In addition, the samples were digested using HACH COD reactor and then analysed using a spectrophotometer (HACH DR3900, United States). Ammoniacal-nitrogen concentration was determined by the Nessler method using a HACH spectrophotometer, as well as TSS and colour. Furthermore, the chemical precipitate was subjected to scanning electron microscopy energy dispersive X-ray (FESEM-EDX), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) to identify the morphology and elemental composition, crystalline structure, and functional groups present in the precipitates, respectively.

RESULTS AND DISCUSSION

pH

In this study, as the concentration of NaOH increased, the pH remained stable within an alkaline range, which is necessary for the formation of insoluble metal hydroxides. At the 30 minutes of reaction, the pH for the conditions of control, 1M, 2.5M and 4.0M were 4.3, 10.95, 11.78, and 11.95, respectively. The stability of the pH ensures that the system remains in the optimal condition for precipitation, preventing the dissolution of the precipitates and facilitating their effective removal. These findings highlight the importance of adjusting the pH to an appropriate alkaline range, as achieved by NaOH addition, to ensure the efficient removal of Sn and Cu, making NaOH a suitable and reliable

agent for chemical precipitation in semiconductor wastewater treatment.

REMOVAL OF TIN AND COPPER

TIN REMOVAL

Figure 1 shows the effectiveness of NaOH at different concentrations (1.0 M, 2.5 M, 4.0 M) in removing Sn from semiconductor wastewater at various time intervals. The control setup (without NaOH) shows a gradual reduction in Sn concentration over time, starting with initial concentration of 3.6 mg/L to 3.0 mg/L after 30 minutes, with a maximum removal of 16.9%. The 1.0 M NaOH concentration significantly improves Sn removal, with the percentage of removal increasing with time, where it reduces to 0.94 mg/L after 30 minutes, reflecting a 68.6% removal. The 2.5 M NaOH concentration performs even better at 30 minutes with removal of 72.4%. The most effective concentration was 4.0 M NaOH, which achieves the highest removal efficiency of 86.8% and the final concentration below the Standard B of Environmental Quality (Industrial Effluent) Regulations 2009. The effectiveness of NaOH precipitation improves with both higher concentrations of NaOH and longer treatment times, with 30 minutes providing optimal removal for all concentrations. Tokuda et al. (2008) investigated Sn precipitation using hydrogen sulphide (H_2S) as a precipitating agent and reported that almost complete precipitation was achieved when the H_2S dosage was equimolar to Sn. More recently, Kaung et al. (2025) reported an innovative approach in which Sn removal reached 89.9% using 50 mg/L polyaluminium chloride (PAC). The removal efficiency increased to 97.5% at 100 mg/L PAC and further improved to 99.8% at 300 mg/L PAC. The precipitation agents can achieve higher removal due to their ability to lower residual metal limits and produce denser, more easily dewaterable sludge. However, NaOH offers distinct advantages over these alternatives owing to its accessibility, low cost, and effectiveness in simultaneously precipitating various heavy metals. Furthermore, kinetic studies demonstrate that NaOH is a highly effective and efficient agent, as evidenced by its high removal.

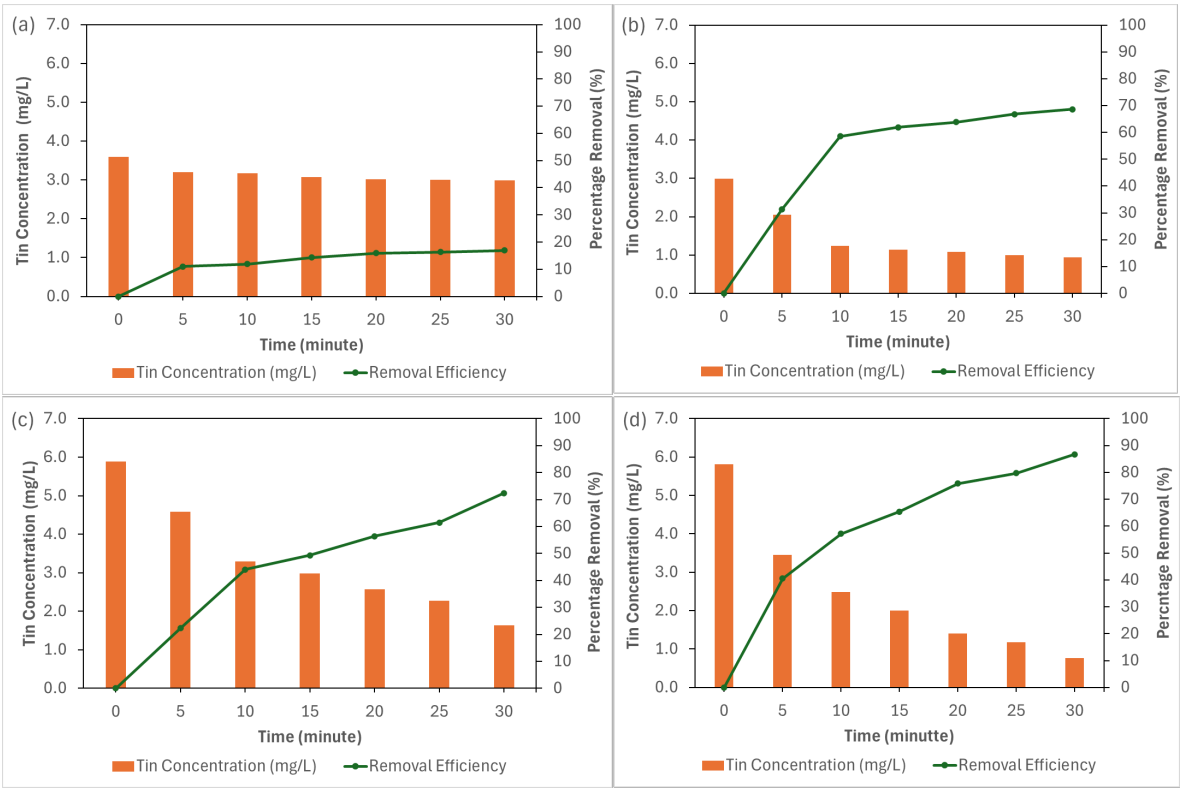


FIGURE 1. Tin concentrations and its removal efficiency under (a) control, (b) 1.0 M NaOH, (c) 2.5 M NaoH, and (d) 4.0 NaOH)

COPPER REMOVAL

Figure 2 presents the effectiveness of NaOH concentrations in removing Cu. The control shows no Cu reduction. The Cu removal increased to 47.9% at 30 minutes reaction as increased the NaOH concentration to 1.0 M. Difference to Sn removal, increased the NaOH to 2.5 and 4.0 M resulted a Cu reduction with removal of 46.3% and 43.9%, respectively. While NaOH supports to reduce Cu concentration, it is not the efficient precipitating agent for Cu removal in the semiconductor wastewater. A study by Chen et al. (2018) investigated Cu removal using three chemical precipitants, namely soda ash (Na_2CO_3), sodium sulfide (Na_2S), and lime ($Ca(OH)_2$). The results showed that Na_2S achieved the highest Cu removal efficiency, exceeding 99.8%, compared to the other precipitants. Abdullah et al. (1999) reported that maximum Cu removal by hydroxide precipitation was achieved within a pH range of 6.5 to 12. Additionally, a study by Ying et al. (2018) found that different treatment methods resulted in varying performances in Pb(II) and Cd(II) removal. The removal efficiency increased when NaOH-calcination-modified kaolin was used, compared to natural kaolin and NaOH-modified kaolin alone. Therefore, future studies may enhance Cu removal from wastewater by applying alternative or combined modification methods.

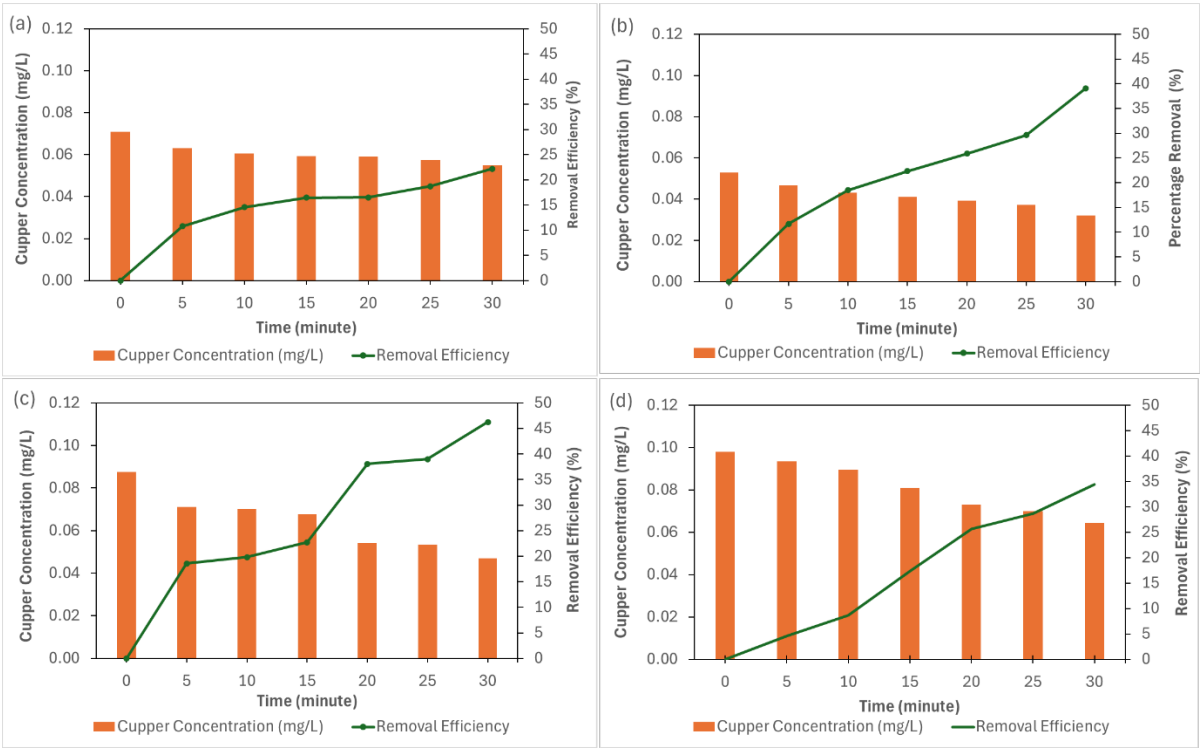


FIGURE 2. Copper concentrations and its removal efficiency under (a) control, (b) 1.0 M NaOH, (c) 2.5 M NaoH, and (d) 4.0 NaOH

CHEMICAL OXYGEN DEMAND AND TOTAL SUSPENDED SOLID REMOVAL

The results show in Figure 3 indicate that chemical precipitation using NaOH effectively reduces COD in the semiconductor wastewater. The removal of COD increased to 43.8% after 30 minutes as the concentration of NaOH increased to 4.0 M. Longer reaction times led to more COD removal, particularly between 20 to 30 minutes reaction. The control sample showed a lower COD reduction of 35.8%, emphasizing that while some natural reduction occurs, chemical treatment significantly enhances the removal of organic pollutants. These findings highlight that higher NaOH concentrations and longer reaction times optimize COD removal, making NaOH precipitation a promising method for treating semiconductor wastewater. Yatim et al. (2020) reported that approximately 53.9% of COD was removed from plating wastewater through hydroxide precipitation at a pH of 8.5. Additionally, the COD result also decreased from 128 mg/L to 59 mg/L for hydroxide precipitation and 12.9 mg/L for sulphide precipitation.

Figure 4 indicates that NaOH significantly reduced TSS, with higher NaOH concentrations and longer treatment times leading to greater removal efficiency. The control sample showed a moderate reduction of 84.6%, while 1.0 M NaOH achieved a maximum removal of 98.5%

after 30 minutes, demonstrating its effectiveness. A 2.5 M and 4.0 M NaOH also showed high removals, but the TSS levels fluctuated slightly after 30 minutes, suggesting a saturation point. Overall, the data highlights that NaOH is highly effective in enhancing COD and TSS removal.

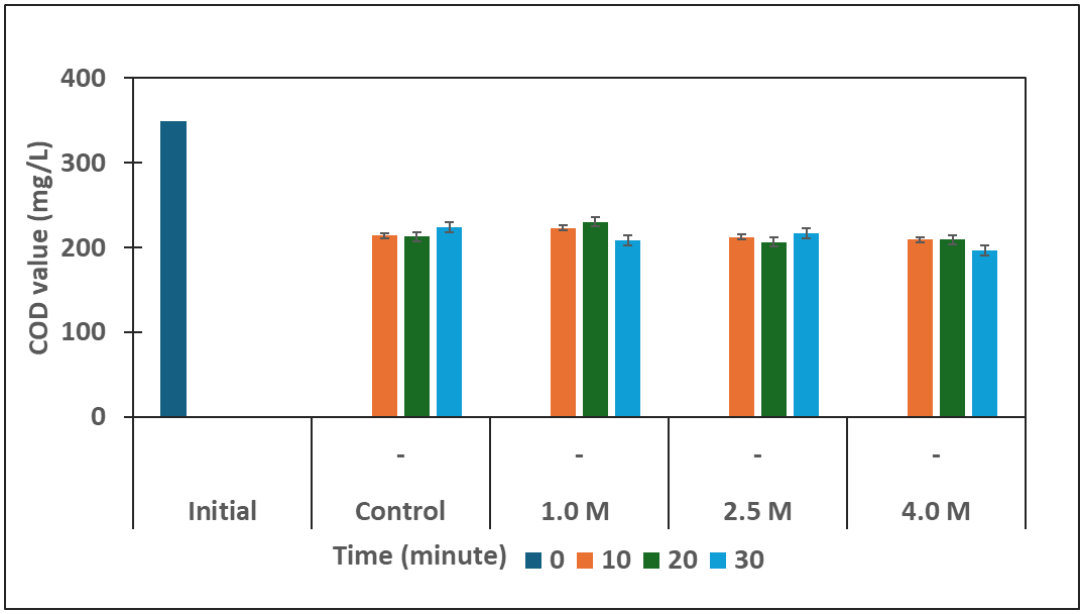


FIGURE 3. Chemical Oxygen Demand reduction

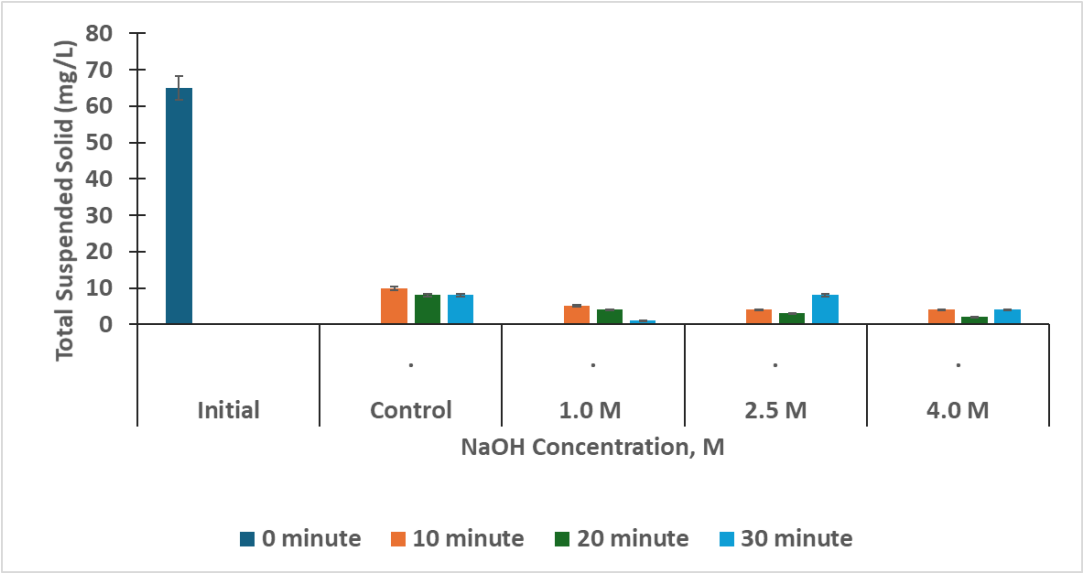


FIGURE 4. Total Suspended Solid reduction

KINETIC FOR THE RATE OF TIN REMOVAL

Understanding the kinetic of Sn removal is essential to determine the rate of process efficiency for the optimization and scaling up of the treatment. A first-order and second-order kinetics was applied to the Sn removal. From the kinetics, it also evaluates the effectiveness of NaOH as a precipitating agent. A first-order reaction means that the rate of removal is directly proportional to the concentration of Sn remaining in the wastewater. As the concentration of Sn decreases, the rate of its removal also decreases. The first-order equation as follows (Adeogun et. al 2021):

$$\ln\left(\frac{C_0}{C_t}\right) = k_1 t \quad (1)$$

where C_0 is the initial concentration of tin, C_t is the concentration of Sn at time t, k_1 is the first-order rate constant, t is the time.

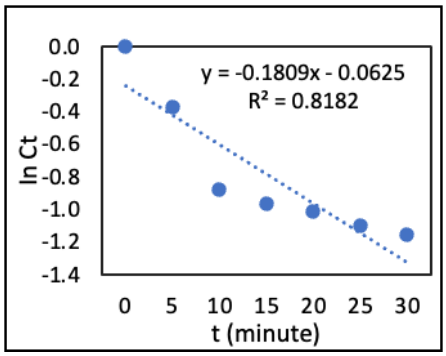
A second-order reaction implies that the rate of removal is proportional to the square of the concentration of Sn in the wastewater. This means that the removal rate decreases more rapidly as the concentration decreases compared to first-order kinetics. The equation for second-order kinetics is (Adeogun et. al 2021):

$$\frac{1}{C_t} - \frac{1}{C_0} = k_2 t \quad (2)$$

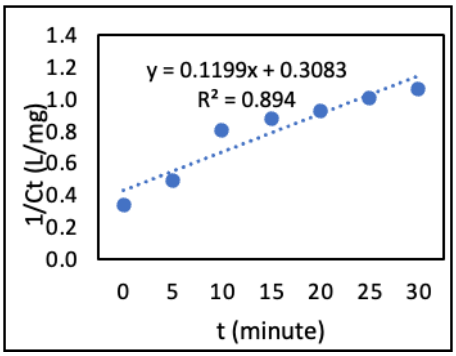
where C_0 is the initial concentration of tin, C_t is the concentration of tin at time t, k_2 is the second-order rate constant, t is the time.

The results show that for the first-order kinetic model, the k_1 increased with the concentration of NaOH, starting from 18.09 min^{-1} at 1.0 M, rising to 24.74 min^{-1} at 2.5 M, and 31.4 min^{-1} at 4.0 M (Table 1). These values demonstrate that the Sn removal process at higher NaOH concentrations fits the first-order model with higher R^2 compared to second-order model (Figure 5). It also indicates a direct relationship between NaOH concentration and the rate of Sn removal. The fact that the k_1 increased with NaOH concentration also suggests that the chemical reaction is dependent on the concentration of Sn the wastewater.

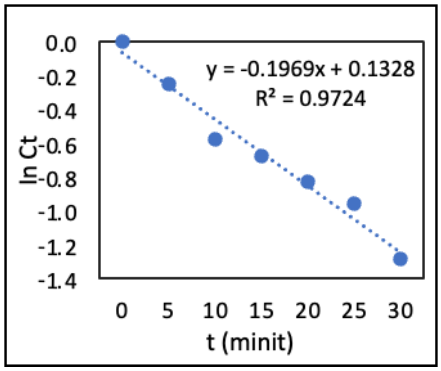
The implications of first-order kinetics for the chemical precipitation process are significant for optimizing wastewater treatment. Since the rate of Sn removal is directly proportional to the concentration of NaOH, increasing NaOH concentration enhances the removal rate, which is crucial for ensuring more efficient and faster treatment. However, it is essential to balance NaOH concentration to avoid excessive chemical use, which could lead to unnecessary costs and waste. Additionally, first-order kinetics suggest that increasing reaction time can improve the removal efficiency, especially when higher concentrations of NaOH are used. This understanding allows for better control of the treatment process, helping to optimize both chemical usage and operational time, ultimately leading to more cost-effective and scalable solutions for the semiconductor wastewater treatment.



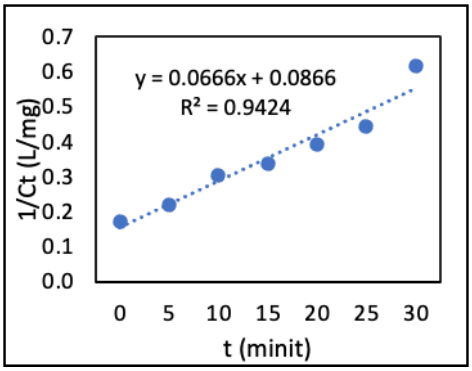
A1



B1



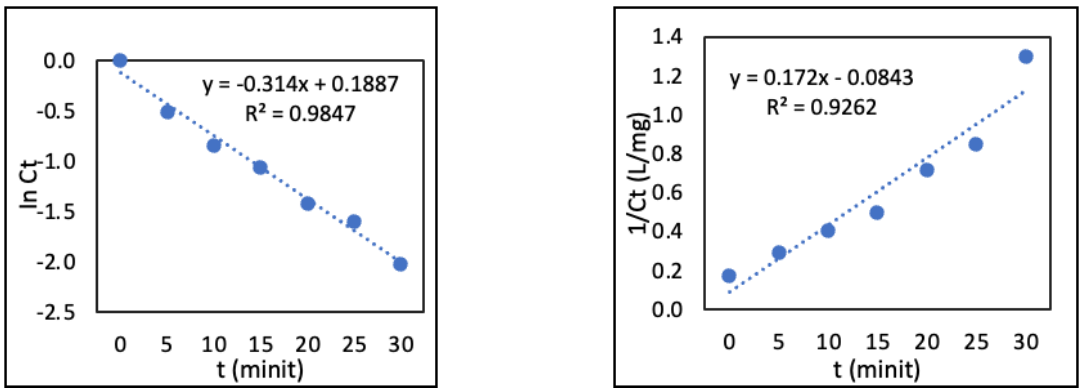
A2



B2

continue...

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A3 B3

FIGURE 5. Plots of (A) first-order, and (B) second-order kinetics

TABLE 1. Kinetic parameters for the first-order and second-order kinetics for Sn removal

Concentration NaOH (M)	First-order		Second-order	
	k_1 (min^{-1})	R^2	k_2 (L.mg^{-1}.min^{-1})	R^2
1.0	18.09	0.8182	11.99	0.8940
2.5	24.74	0.9724	6.66	0.9424
4.0	31.40	0.9847	17.2	0.9262

CHARACTERIZATION OF PRECIPITATE

MORPHOLOGICAL AND ELEMENTAL EVALUATION

FESEM was used to examine the surface morphology of the precipitate formed after chemical precipitation using NaOH. The surface morphology of the precipitate showed a rough and textured structure, with small particles forming aggregates and fine fibrous networks (Figure. 6a). The EDX spectrum (Figure 6b) shows the dominant elemental composition of the precipitate. The EDX spectrum indicates that Sn was the most abundant element, with a weight percentage of 56.7%. Other elements include oxygen (O), carbon (C), silicon (Si), copper (Cu), phosphorus (P) and magnesium (Mg).

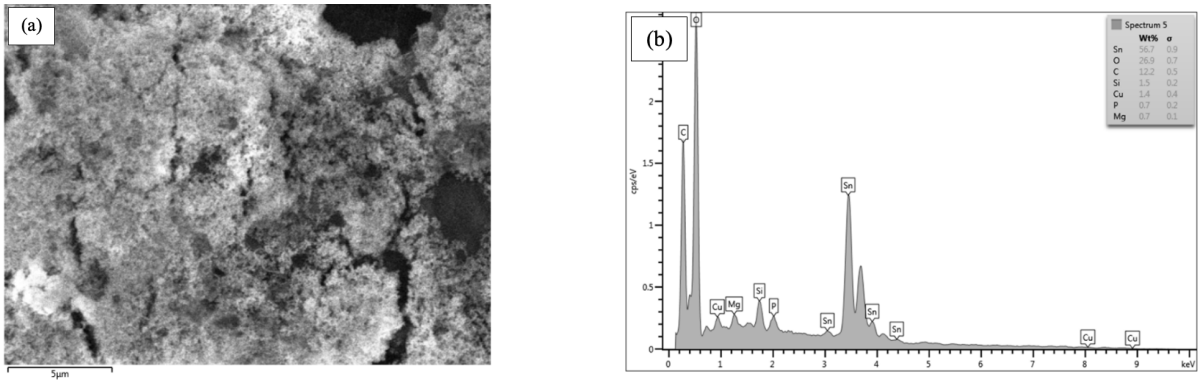


FIGURE 6. (a) Surface morphology of the precipitate, (b) EDX spectrum elemental analysis

FUNCTIONAL GROUP OF THE PRECIPITATE

FTIR analysis was conducted to identify the chemical functional group of the precipitate obtained from the NaOH chemical precipitation process. Figure 7 presents the FTIR spectrum of the precipitate, indicating absorption bands at various wavelengths associated with the chemical bonds in metal hydroxides, providing insights into the chemical composition of the formed precipitate. Peak at 3,290 cm^{-1} indicates the presence of hydroxyl groups (-OH) stretching, commonly found in metal hydroxides such as $Cu(OH)_2$ and $Sn(OH)_2$. According to Pramanik et al. (2014), the peaks at 3,564 and 3,311 cm^{-1} correspond to the stretching vibrations of -OH group in $Cu(OH)_2$. Its presence confirms the formation of metal hydroxides. Metal-oxygen stretching at 1,637 and 1,036 cm^{-1} is associated with the vibration of metal-oxygen bonds in metal hydroxides, indicating that the heavy metals have been precipitated as metal hydroxides. Wavelength at 526 cm^{-1} reflects to metal-ligand bond vibrations in hydroxide complexes or metal salts left in the precipitate. Reported by Naveenkumar et al. (2018), peaks at 628 and 520 cm^{-1} associated with streching vibrations of the Cu-O bands. The FTIR results showed that precipitation using NaOH successfully formed metal hydroxides $Cu(OH)_2$ and $Sn(OH)_2$, providing clear evidence of the chemical composition of the precipitate and supports assess the effectiveness of the chemical precipitation method used.

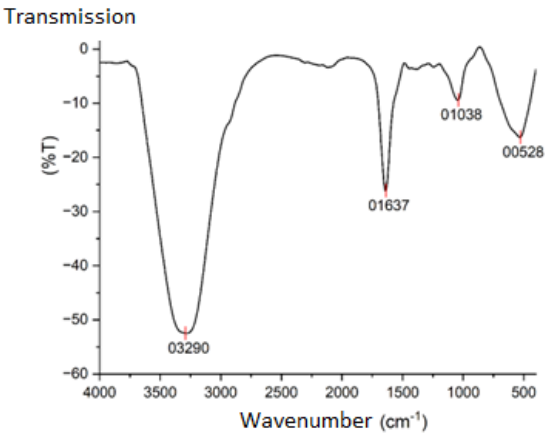


Figure 7. FTIR spectrum of the chemical composition of heavy metal precipitate

CRYSTAL STRUCTURE OF PRECIPITATE

XRD was used to determine the crystal structure of precipitate formed from the chemical precipitation. Figure 8 shows the spectrum of the XRD analysis performed on the sample. The XRD graph shows a sharp main peak at $2\theta = 22.5°$, indicating the presence of an ordered crystal structure in the sample. This peak suggests that the primary component in the sample is $Sn(OH)_2$, with a clear and stable crystal arrangement. The high intensity of the peak further confirms that $Sn(OH)_2$ crystals are successfully produced.

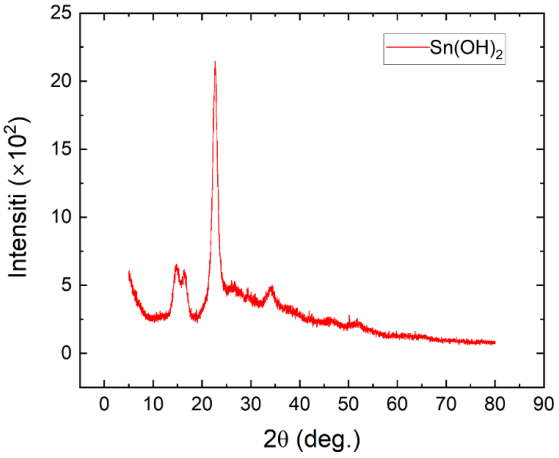


FIGURE 8. XRD spectrum of precipitate

CONCLUSION

The increasing demand for the semiconductor industry globally has led to a significant rise in water usage during production processes, subsequently increasing semiconductor wastewater generation. Untreated and uncontrolled discharge of this wastewater containing heavy metals can harm the environment due to its toxicity. This study utilized NaOH as chemical precipitation agent as a primary method for Sn and Cu removal from the wastewater. NaOH serves as the key precipitating agent due to its accessibility, low cost and effectiveness in removing various heavy metals. The study concluded that increasing the concentration of NaOH and extending the settling time enhanced the removal of Sn and Cu, achieving 86.8% and 46.3% removal, respectively, within 30 minutes. The kinetic analysis revealed that Sn removal followed a first-order reaction model, with the rate constant increasing at higher NaOH concentrations. This indicates that the rate of Sn removal is directly proportional to its concentration in the wastewater. Furthermore, the $Sn(OH)_2$ precipitate formed during the NaOH treatment process shows potential for reuse in various applications, including materials manufacturing, surface engineering, and chemical synthesis.

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DECLARATION OF COMPETING INTEREST

None.

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