# Determining the Effect of Nickel and Cadmium Ions in Struvite Crystallization Process

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***Abstract*— Struvite is an eco-friendly fertilizer widely produced from waste streams. Landfill leachate, rich in ammonium-nitrogen ($NH_4$-N), encourages the recovery of $NH_4$-N in the form of struvite. However, the presence of heavy metals, particularly cadmium (Cd) and nickel (Ni), may affect the purity of struvite crystals, thereby hindering its applicability as a fertilizer. While previous studies have examined the effects of various heavy metals on struvite crystallization, the specific impact of Cd and Ni, especially in landfill leachate, remains insufficiently studied. This study aimed to fill this gap by investigating the effect of Cd and Ni on the purity and morphology of struvite crystals obtained from synthetic solutions (Phase 1) and synthetic landfill leachate (Phase 2). Additionally, a kinetics study was conducted to determine the fate of Ni and Cd during struvite crystallization. Batch experiments of struvite precipitation were performed with varying concentrations of Cd and Ni. Aqueous analysis results indicated a minor reduction in $NH_4$-N recovery in both phases when Cd or Ni were present. Furthermore, XRD and SEM analyses of solid samples demonstrated that all crystals were highly pure struvite. The impact of Cd and Ni in Phase 2 was slightly more significant than in Phase 1. The kinetics study suggested that struvite can be safely recovered from landfill leachate contaminated with Cd and Ni. However, further research is needed on actual landfill leachate to fully understand the combined and advanced effects of heavy metals on struvite crystallization.**

***Index Terms*— Struvite, landfill leachate, cadmium, nickel, ammonium-nitrogen**

## I. Introduction

The process of precipitation of ammonium nitrogen ($NH_4$-N) forming magnesium ammonium phosphate ($MgNH_4PO_4.6H_2O$), known as Struvite, has been deeply studied since the 18th century. Struvite crystallization technology was developed as a phenomenal process for the purpose of phosphate and nitrogen recovery from waste streams [1], since it can be applied to a wide range of wastewaters containing high concentrations of nitrogen [2]. The major advantage of this technique is the production of struvite; a slow-release fertilizer that is environmentally friendly and safe for plants and soil [3]. This is crucial to assist planners and decision makers to accomplish agricultural practice that comply with the social, environmental and economic strategies [4].

Wastewater frequently contains various compounds and elements that can interfere with struvite crystallization, including heavy metals, which pose a significant threat to the purity of the formed struvite [5], [6]. According to Huang et al. [7], heavy metals concentrations in struvite increases linearly with their original amounts in wastewater. In addition, heavy metal co-precipitation may impact struvite crystal formation, phase composition, shape, and surface chemistry of struvite precipitates [6]. These impacts could hinder the application of struvite as a fertilizer [8]. A recent study has shown that the plant's capability to translocate heavy metals from landfill leachate into the plant tissue is very alarming [9]. The effect of a number of heavy metals on struvite crystallization, such as copper (Cu) and zinc (Zn) [10], chromium (Cr) [11], [12] and arsenic (As) [13], has been widely studied. However, to the best of the authors' knowledge, the effect of Ni and Cd on struvite crystallization was not investigated before, although they are highly concentrated in different types of wastewaters, specifically in landfill leachate [14], [15].

On the other hand, struvite has been successfully crystallized in heavy metals-contaminated waste streams, such as semiconductor wastewater [16], anaerobically digested chicken slurry [17], urine [18] and swine wastewater [19]. Therefore, the main goal of this study is to investigate the fate and kinetics of Ni and Cd ions during struvite crystallization in landfill leachate.

## II. Methodology

This study consists of 3 experimental phases. In Phase 1, a synthetic solution of ammonium chloride ($NH_4Cl$) was used for struvite crystallization, while in Phase 2, synthetic wastewater was used with the same concentration of struvite components and heavy metals as Phase 1. Phase 3 focused on the kinetics of struvite crystallization under different concentrations of Ni and Cd. All used reagents were of analytical grade.

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### A. Phase 1: Struvite Crystallization Using Synthetic Solution

The synthetic solution of 0.07M $NH_4Cl$ was prepared representing the normal range of $NH_4$-N in landfill leachate (around 990 mg/L). Equimolar ratio of all struvite components (i.e., Mg, N and P) was implied in this study, hence the same molar value was used for $PO_4$ and Mg. Due to its high solubility in water, magnesium sulphate heptahydrate ($MgSO_4.7H_2O$) was used as the Mg source, while sodium dihydrogen phosphate ($NaH_2SO_4$) was used as the source of $PO_4$. All stock solutions were prepared using deionized water and the same stock solutions were used throughout the study to obtain uniform and precise results. With regards to heavy metals solution as shown in Table I. For Ni and Cd sources, nickel (II) chloride hexahydrate ($NiCl.6H_2O$) and cadmium chloride hemi pentahydrate ($CdCl.2½H_2O$) were applied, respectively. The concentration ranges of Cd and Ni were chosen based on the rates commonly found in actual landfill leachates.

TABLE I
CONCENTRATIONS OF NICKEL AND CADMIUM IN PHASE 1 AND 2

| Heavy metal | Molar concentration (µmol/L) |
|---|---|
| Ni | 8.54 |
| | 38.87 |
| | 58.10 |
| Cd | 4.38 |
| | 21.90 |
| | 43.79 |

The initial pH was adjusted using 1.0M sodium hydroxide (NaOH) solution until the pH is 8.0±0.05, before the reaction solution was stirred for 30 min using magnetic stirrer. Consequently, the sample was left to settle down for 1 hr. Liquid samples were collected using 0.45 µm syringe filters for further analyses, whereas the remaining solution was filtered using vacuum filter, then washed twice with deionized water before drying for 24 hours in room temperature. Finally, the solid samples were collected for further analysis.

### B. Phase 2: Struvite Crystallization Using Synthetic Landfill Leachate

The same experiment was repeated for Phase 2, with the replacement of synthetic solution by synthetic landfill leachate. The composition of the synthetic landfill leachate was adapted from Rowe et al. [20] (Table II). The same molar concentrations of Mg, $NH_4$ and $PO_4$, as well as concentrations of Ni and Cd, applied in Phase 1 were used during the preparation of synthetic wastewater in Phase 2.

TABLE II
COMPOSITION OF SYNTHETIC LANDFILL LEACHATE

| Component | Per 1 L |
|---|---|
| Acetic acid (100%) | 5 mL |
| Propionic acid (100%) | 1 mL |
| Butyric acid (100%) | 1 mL |
| $MgSO_4.7H_2O$ | 17.253 g |
| $Na_2CO_3$ | 0.120 g |
| $Ca(NO_3)_2$ | 0.100 g |
| $NH_4Cl$ | 3.744 g |
| $NaH_2SO_4$ | 8.398 g |

### C. Phase 3: Kinetics Study of Struvite Crystallization

A series of struvite crystallization samples were prepared using the synthetic wastewater, with a working volume of 500 mL and Mg: N:P molar ratio of 1:1:1, with different initial concentrations of Ni or Cd, independently (as shown in Table III). The concentration of Ni and Cd was selected as the lowest and the highest concentration of the typical one in landfill leachate (as a type of wastewater that usually contains high loads of both $NH_4$-N and heavy metals).

Adjustment of initial pH was performed similarly to Phase 1 but reached an initial pH of 7.5±0.02 instead. After that, the mixture was stirred for 1 hour. Along with the mixing, 2 mL aqueous samples were collected from the beakers for reaction times of 1, 3, 5, 15, 30 and 60 minutes, using a syringe filter. Besides, pH was measured every 1 min to maintain the initial pH of the solution. Any adjustment of pH was made by adding 1.0M NaOH solution. This step was repeated for an initial pH of 8.5. So, for each pH, the total number of samples for kinetic study equaled 5.

TABLE III
INITIAL CONCENTRATIONS OF NICKEL AND CADMIUM IN KINETICS STUDY

| Run | pH | Heavy metal | Heavy metal initial concentration |
|---|---|---|---|
| 1 | 7.5 | Blank | 0 |
| 2 | | Cd | 0.5 |
| 3 | | | 10.0 |
| 4 | | Ni | 0.5 |
| 5 | | | 13.81 |
| 6 | 8.5 | Blank | 0 |
| 7 | | Cd | 0.5 |
| 8 | | | 10.0 |
| 9 | | Ni | 0.5 |
| 10 | | | 13.81 |

The kinetics order used was the first-order kinetics model as stated in Equation (1) because it is the best fit model to determine the kinetics parameter value (k) of struvite reaction [21]. In addition, the kinetics of struvite were calculated at two different pH values (7.5 and 8.5), as pH is a major influencing parameter in the kinetics study [15]. The concentration of Mg is expected to change over time, and the kinetic constants of struvite crystals were estimated using Equation (1):

$$\ln(C - C_{eq}) = -kt + \ln(C - C_0) \quad (1)$$

where $C$ is the concentration of Mg at any time in moles (M), $C_{eq}$ is the concentration of Mg at equilibrium in moles (M), $C_0$ is the initial concentration of Mg (M) at time = zero (t = 0), $k$ is the reaction rate constant in min-1 and $t$ is crystallization time in minutes (min).

### D. Analysis of Samples

The concentration of $NH_4$-N in liquid samples was measured using Nessler's standard method, while $PO_4$ was measured using spectrophotometry analysis (HACH DR6000). Atomic adsorption spectrophotometry (AAS, 3300, Perkin Elmer, USA) was used to analyze the concentrations of Ni and Cd in liquid samples. Solid samples of struvite precipitates were analyzed using X-ray diffraction (XRD, Rigaku Americas Corporation, The Woodlands, TX) and Scanning Electron Microscopy (SEM, Gemini, Zeiss Supra Series, Germany).

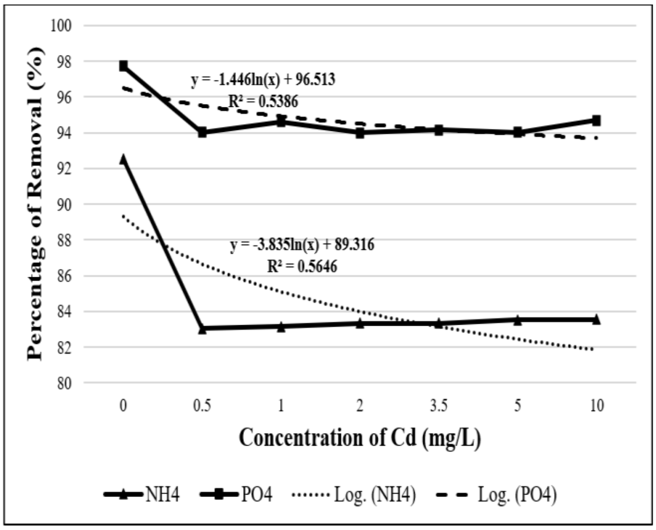
## III. Results and Discussion

### *A. Removal of $NH_4$-N and $PO_4$ in the presence of Cd and Ni*

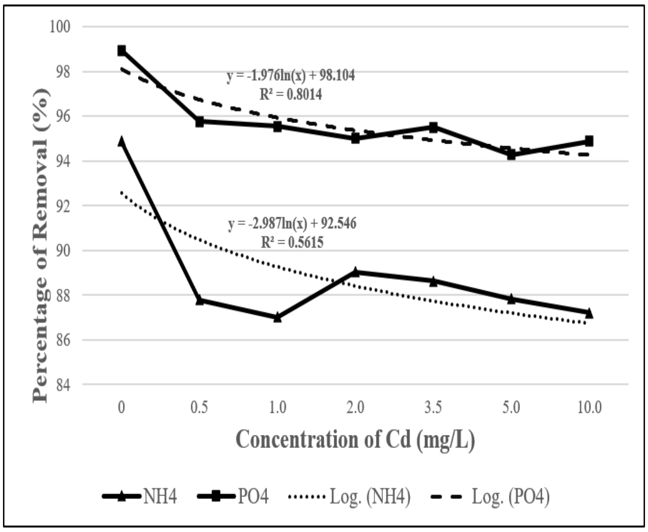
Fig. 1 shows the removal percentage of $NH_4$-N and $PO_4$ when interacting with Cd and Ni. For Phase 1, as illustrated in Fig. 1(a) and (c), the removal percentage for both $NH_4$-N and $PO_4$ was 92.56% and 97.74%, respectively. Meanwhile, for Phase 2, as illustrated in Fig. 1(b) and (d), the removal percentages witnessed a minute change; removal of $NH_4$-N and $PO_4$ was 94.88% and 98.93%, respectively. This change is expected as the characteristics of precipitation mediums in Phase 1 and Phase 2 are different. Furthermore, the interference with the organic content or trace metals contained in the synthetic landfill leachate may cause such nuisance in the readings [19]. Even though, the results are consistent with previous studies; Huang et al. [22] achieved 92% removal of $NH_4$-N in landfill leachate, and Miroslav et al. [13] achieved 96% removal of $PO_4$ in industrial wastewater by struvite crystallization.

### *B. Investigation of Precipitates' Morphology and Composition*

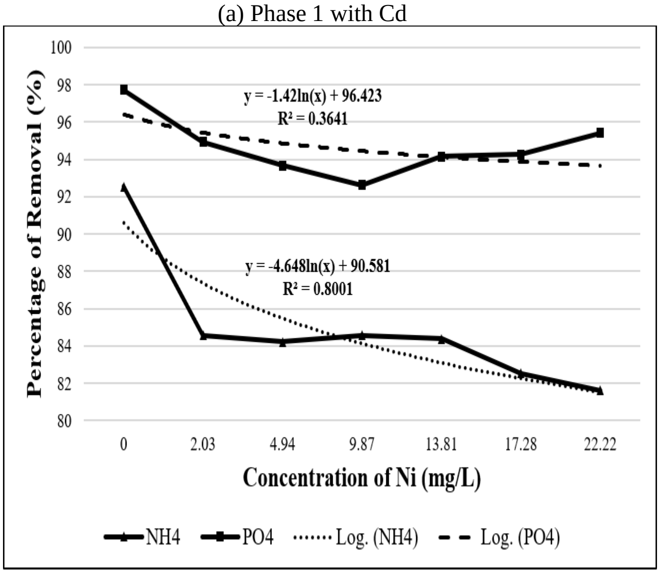
To verify the effect of Cd and Ni on struvite crystals' morphology, XRD analysis was conducted on solid precipitates. The results obtained for all solid samples showed high matching with struvite XRD standard pattern (Fig. 2 (a) and (b)) and all the solid samples were in crystal form. However, the samples' pattern displayed some background noise that could be due to amorphous compounds associated with the heavy metals' ions of $Ni^{2+}$ and $Cd^{2+}$. In general, the XRD patterns demonstrated that the main composition of the precipitates was struvite. Similar findings were reported by Huang et al. [7] who studied the interaction of Zn, Cr and Cu with struvite crystallization in synthetic swine wastewater. This means that the behaviors of these heavy metals in struvite crystallization process are interestingly alike.



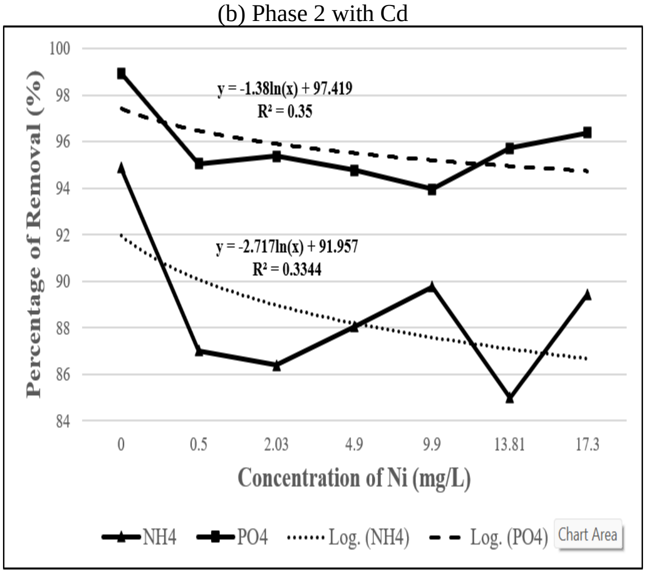

(a) Phase 1 with Cd




(b) Phase 2 with Cd




(c) Phase 1 with Ni




(d) Phase 2 with Ni

Fig. 1. Removal of $NH_4$-N and $PO_4$ in Phase 1 (a & c) and Phase 2 (b & d).

*C. Investigation of Precipitates Morphology and Composition*

To verify the effect of Cd and Ni on struvite crystals' morphology, XRD analysis was conducted on solid precipitates. The results obtained for all solid samples showed high matching with struvite XRD standard pattern (Fig. 2 (a) and (b)) and all the solid samples were in crystal form. However, the samples' pattern displayed some background noise that could be due to amorphous compounds associated with the heavy metals' ions of $Ni^{2+}$ and $Cd^{2+}$. In general, the XRD patterns demonstrated that the main composition of the precipitates was struvite. Similar findings were reported by Huang et al. [7] who studied the interaction of Zn, Cr and Cu with struvite crystallization in synthetic swine wastewater. This means that the behaviors of these heavy metals in struvite crystallization process are interestingly alike.

Struvite precipitates were further characterized using SEM images. The analysis included struvite samples obtained from the lowest and highest concentrations of Cd and Ni in both Phase 1 and Phase 2, as well as a control sample. Fig. 3-6 display SEM images of selected precipitates from Phase 1 and Phase 2. The SEM images of the control sample from Phase 2 exhibit a distinct crested surface and numerous gullies, with a more prismatic shape compared to the control sample from Phase 1. This difference could be attributed to variations in the composition of the reaction solutions between the phases (i.e., synthetic solution versus synthetic landfill leachate). The reaction solution in Phase 2 contains organic acids that may account for these observed differences. This hypothesis is consistent with the findings of Tang et al. [23], who utilized synthetic solutions to produce struvite crystals.

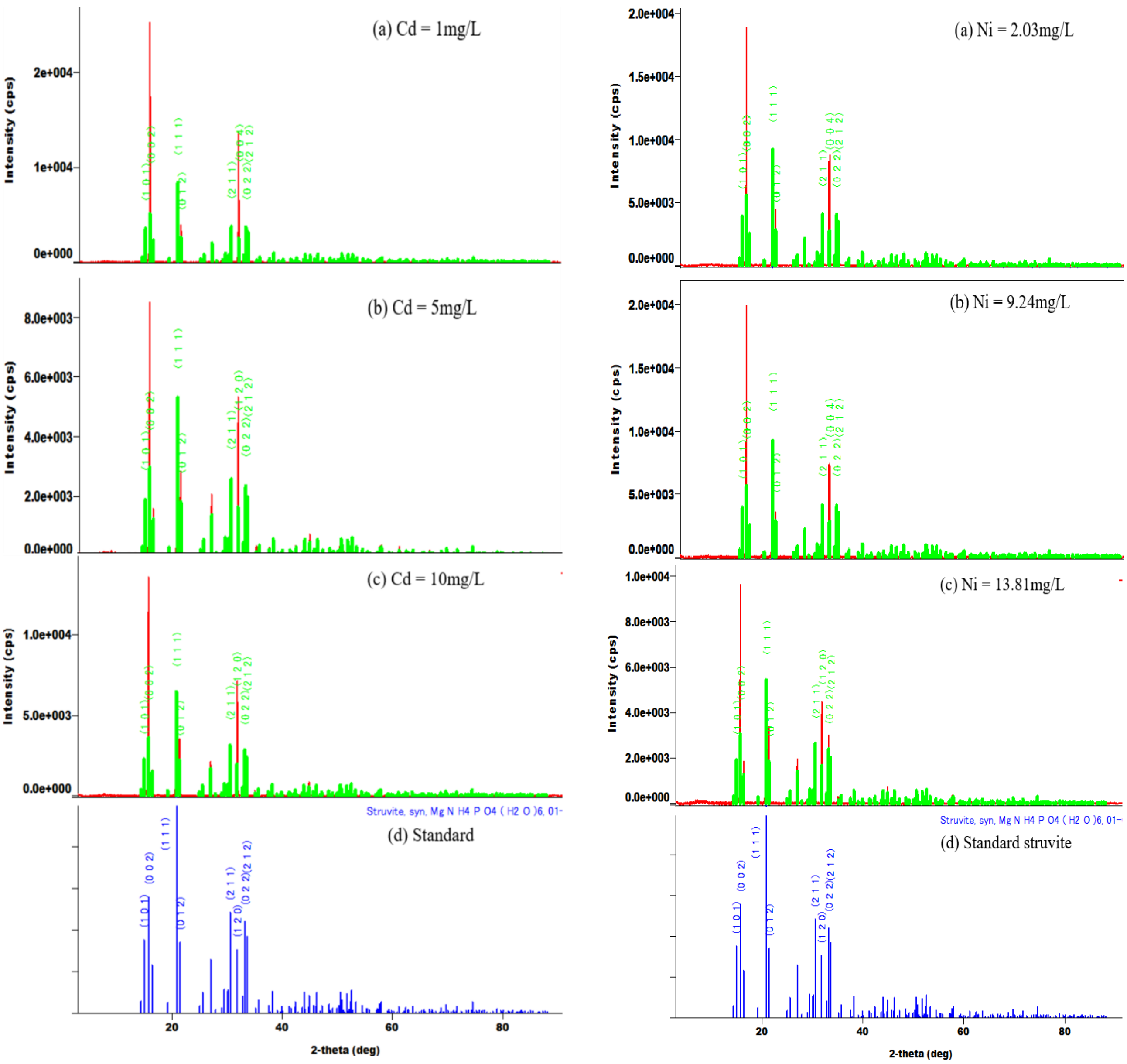
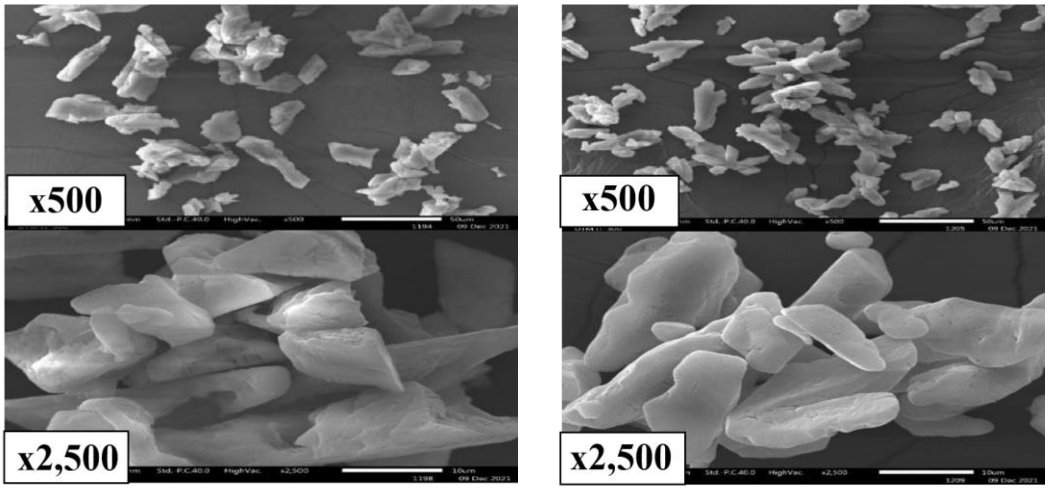



Fig. 2. XRD analysis of solid samples from Phase (1) in the presence of Cd and Ni.

Fig. 3 and 4 depict the influence of Cd and Ni on the shape of struvite crystals in Phase 1. It is evident that low concentrations of Cd did not significantly alter the shape or surface of struvite crystals. Even at higher concentrations of Cd (10 mg/L), only a smoother surface was observed, indicating minimal impact on struvite morphology when using synthetic solution. This finding corroborates with earlier spectrometry analyses of Cd-contaminated struvite. In contrast, Ni caused distinct holes on the crystal surfaces, irrespective of Ni concentration levels, suggesting a more significant effect on struvite shape and morphology.

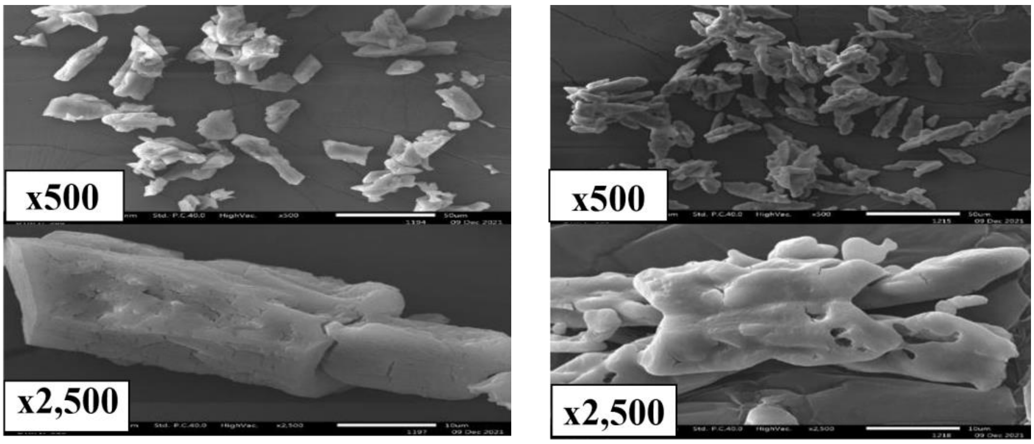
In Phase 2, Cd and Ni exhibited a more noticeable effect on struvite crystals. According to Fig. 5 and 6, struvite crystals with X-shaped structures and blunt, round edges were observed with Cd, while Ni resulted in several holes on the crystal surfaces. These observations are consistent with previous studies by Manzoor et al. [24] and Kemacheevakul et al. [25], indicating an enhanced effect of Cd and Ni in synthetic landfill leachate, potentially due to interactions with organic acids and other pollutants present in the solution.

Overall, as discussed later, struvite can be safely recovered from waste streams contaminated with Cd and Ni within the examined ranges. Wen et al. [5] demonstrated the cultivation of cabbage using struvite contaminated with heavy metals recovered from human urine and municipal wastewater. The detected heavy metal levels in the vegetables were below Chinese food safety standards, suggesting that even when struvite incorporates heavy metals, it can still be safely used in agriculture.




**Blank** **Cd = 10 mg/L**

Fig. 3. SEM images of struvite from blank and Cd contaminated solution (Phase 1).




**Blank** **Ni = 13.81 mg/L**

Fig. 4. SEM images of struvite from blank and Ni contaminated solution (Phase 1).

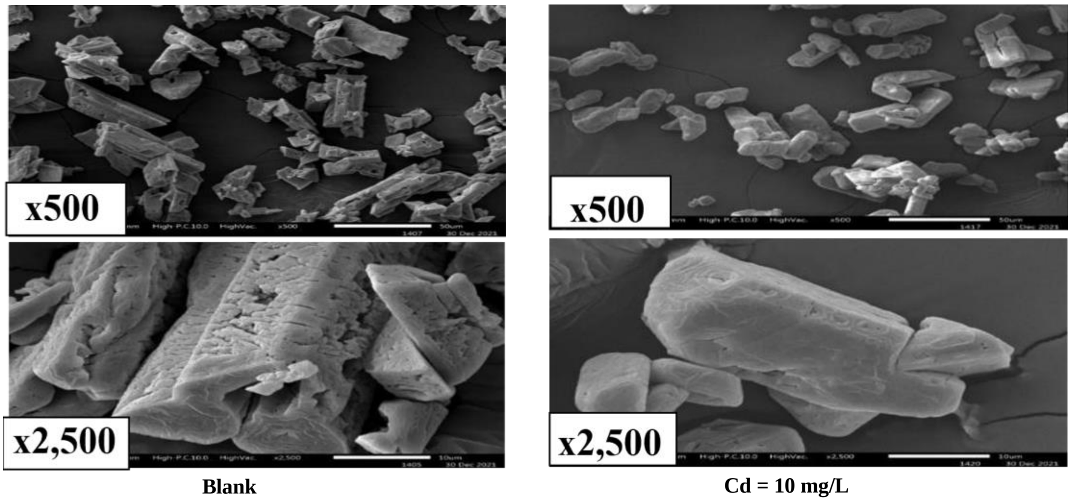



Fig. 5. SEM images of struvite from blank and Cd contaminated solution (Phase 2).

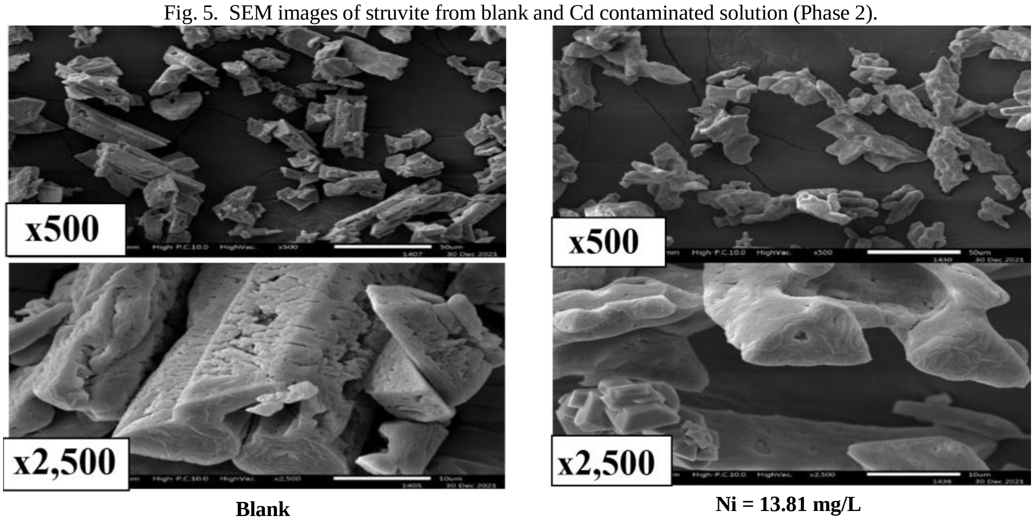



Fig. 6. SEM images of struvite from blank and Ni contaminated solution (Phase 2).

*D. Kinetics of struvite crystallization in the presence of Ni and Cd*

Kinetics of struvite crystallization in the presence of Cd and Ni was determined under pH 7.5±0.2 and 8.5±0.2 using the synthetic landfill leachate solution. As mentioned earlier, kinetics constant (k) for struvite crystallization was calculated using Equation (1). To confirm the results obtained in this study and how it matched with the first-order kinetic model *ln (C – Ceq)* versus time for each pH value was plotted. A straight line was then outlined for each Cd and Ni concentration to determine the regression equation and correlation coefficient (R). Data with R value higher than 0.70 was fit and strongly correlated, as presented in Table IV. All correlation coefficients are more than 0.70, showing a strong correlation between parameters and validating the data obtained in this kinetics study.

In this study, kinetic constants were expected to change with varying pH levels and initial metal concentrations. When comparing the kinetic constants at different initial concentrations of Cd and Ni at pH 7.5, the kinetic constant increased with the increase in the initial concentration of both Cd and Ni. The same pattern was observed for the interaction of struvite with Cd at pH 8.5. However, a different pattern was reported by Perwitasari et al. [26], where the kinetic constants of $Cu^{2+}$, $Pb^{2+}$, and $Zn^{2+}$ decreased as the initial heavy metal concentration increased. The distinct pattern of kinetic constants found in this study could be attributed to the complexity of the synthetic wastewater compositions used.

TABLE IV
KINETICS CONSTANT (K) OF STRUVITE IN PRESENCE OF CADMIUM AND NICKEL

| pH | Concentration (mg/L) | Regression equation | Correlation coefficient (R) | k ($hr^{-1}$) |
|---|---|---|---|---|
| 7.5 | 0 (Blank) | y = -0.0746x +2.08 | 0.8154 | 4.476 |
| | Cd = 0.50 | y = -0.1221x +0.79 | 0.9706 | 7.326 |
| | Cd = 10.00 | y = -0.3576x +1.53 | 0.8967 | 21.456 |
| | Ni = 0.50 | y = -0.2106x + 0.62 | 0.8086 | 12.636 |
| | Ni = 13.81 | y = -0.3369x + 0.66 | 0.8596 | 20.214 |
| 8.5 | 0 (Blank) | y = -0.1069x + 1.83 | 0.8136 | 6.414 |
| | Cd = 0.50 | y = -0.2228x + 0.30 | 0.7417 | 13.368 |
| | Cd = 10.00 | y = -0.3014x + 0.94 | 0.8074 | 18.084 |
| | Ni = 0.50 | y = -0.3614x + 0.83 | 0.8130 | 21.684 |
| | Ni = 13.81 | y = -0.1068x + 1.60 | 0.8037 | 6.408 |

Meanwhile, during pH 8.5, the interaction of struvite crystallization with Ni showed that the kinetics constant decreases as the initial concentration increases. For an initial Ni concentration of 13.81 mg/L, the kinetics constant is lower than that of the blank struvite. Additionally, in Phases 1 and 2, at this specific Ni concentration, XRD analysis detected a few impurities. One of the impurities is magnesium tetra- ammonium cyclotriphosphate tetrahydrate

($Mg(NH_4)_4(P_3O_9)_2.4H_2O$), which represents another phase of struvite [27]. The co-precipitation of these impurities could explain the low kinetics constant of struvite at this concentration at pH 8.5.

## IV. Conclusions

The recovery of $NH_4$-N from waste streams in the form of struvite is widely applied to various types of wastewaters, such as landfill leachate. However, heavy metals in landfill leachate could restrict the use of the obtained struvite, as they can attach to struvite crystals. The results of this study illustrate that Cd and Ni (within the investigated concentrations) have a minor effect on the purity and morphology of struvite, as verified by AAS, XRD, and SEM analyses. In conclusion, although the effects of Cd and Ni were found to be insignificant, further studies are necessary to explore the impact of heavy metals on struvite in actual waste streams.

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