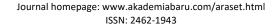


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Investigation on the Effect of Nickel and Cadmium on Struvite Crystallization in Landfill Leachate

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ABSTRACT

Struvite is an eco-friendly fertilizer that is widely produced from waste streams. Landfill leachate is rich in ammonium-nitrogen (NH₄-N), which encourages its recovery in the form of struvite, however, the presence of heavy metals, especially Cd and Ni, could affect the purity of struvite crystals. This work aimed at studying the effect of Cd and Ni on the purity and morphology of struvite crystals obtained from synthetic solution (Phase 1) and synthetic landfill leachate (Phase 2). Batch experiments of struvite precipitation were implemented with different concentrations of Cd and Ni. The results of aqueous analysis showed minor reduction in NH₄-N recovery in both phases when Cd or Ni existed. Moreover, XRD and SEM analyses of solid samples illustrated that all crystals were highly pure struvite. In addition, the effect of Cd and Ni in Phase 2 was slightly more significant than in Phase 1. The results suggested that struvite can be safely recovered from landfill leachate contaminated with Cd and Ni, however, more intense research should be conducted on actual landfill leachate to have deeper understanding of mutual and advanced effects of heavy metals.

Keywords:

Struvite; landfill leachate; cadmium; nickel; ammonium-nitrogen

1. Introduction

Landfills pose serious threat to the environment due to the hazardous leachates produced. Landfill leachate is a result of complicated chemical reactions within the solid wastes components, which make it highly concentrated in ammonium nitrogen (NH4-N), organic content and some heavy metals [1]. Several techniques were introduced for the treatment of landfill leachate, such as activated carbon [2], ammonia stripping [3] and chemical precipitation [4]. On the other hand, the sustainable practice of solid waste management promotes the recovery of valuable products from leachate like food waste composting [5,6], biogas [7] and organic fertilizers [8]. In general, recycling practice promotes environmental conservation, limits pollution, and contributes to circular economy benefits [9-11].

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The process of precipitation of ammonium nitrogen (NH₄-N) forming magnesium ammonium phosphate (MgNH₄PO₄.6H₂O), known as Struvite, has been deeply studied since 18th century. Struvite crystallization technology was developed as a phenomenal process for the purpose of phosphate and nitrogen recovery [12], since it can be applied to a wide range of wastewaters containing nitrogen in the form of NH₄-N [13]. The major advantage of this technique is the recovery of NH4-N in the form of struvite; a slow-release fertilizer that is environmentally friendly and safe for plants and soil [14]. Furthermore, struvite crystallization process contributes significantly to the circular economy concept of wastewater treatment plants, via recovery of phosphate from specific units in the plant, such as digester recirculation pumps and activated sludge storage tanks [15].

Wastewater often comprises different kinds of components, such as organic material and heavy metals, which may interfere with struvite precipitation. This is considered a significant threat to the purity of the formed struvite, which could hinder the application of struvite as a fertilizer [16]. According to Huang *et al.*, [17], heavy metals accumulation in struvite increases linearly with their original concentrations in the wastewater. In addition, heavy metals co-precipitation may affect the formation, content, morphology, and surface chemistry of struvite precipitates [18]. Moreover, struvite contaminated with heavy metals may possess negative impact on plants. A recent study has shown that the plant's capability to translocate heavy metals from landfill leachate into the plant tissue is very alarming [19]. The effect of a number of heavy metals on struvite crystallization, such as copper (Cu) and zinc (Zn) [20], chromium (Cr) [21] and arsenic (As) [22], 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, especially landfill leachate [23, 24].

Landfill leachate is the waste stream generated from municipal or industrial landfills, which contains extremely high concentrations of NH₄-N (exceeding 1,000 mg/L), as well as other pollutants [25, 26]. This encourages the recovery of NH₄-N in the form of struvite. However, the presence of heavy metals could be a threat to the purity of struvite, which might hinder its further application in agronomic field. Therefore, this study aimed to investigate the effect of Ni and Cd on struvite crystals obtained from landfill leachate.

2. Methodology

This study consists of 2 experimental phases. In Phase 1, a synthetic solution of ammonium chloride (NH_4CI) was used for struvite crystallization, while in Phase 2, synthetic landfill leachate was used with the same concentration of struvite components and heavy metals as Phase 1. All used reagents were of analytical grade.

2.1 Phase 1: Struvite Crystallization Using Synthetic Solution

The synthetic solution of 0.07M NH₄Cl was prepared representing the normal range of NH₄-N in landfill leachate (around 990mg/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 PO₄ and Mg. Due to its high solubility in water, magnesium sulphate heptahydrate (MgSO₄.7H₂O) was used as the Mg source, while sodium dihydrogen phosphate (NaH₂SO₄) was used as the source of PO₄. 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 contamination, certain amount of Ni and Cd solutions were spiked into the synthetic solution as shown in Table 1. For Ni and Cd sources, nickel (II) chloride hexahydrate (NiCl.6H₂O) and cadmium chloride hemi pentahydrate (CdCl.2½H₂O) were

applied, respectively. The concentration ranges of Cd and Ni were chosen based on the rates commonly found in actual landfill leachates [27,28].

Struvite precipitation normally occurs at pH values range from 7.0 to 9.50 [13]. A pH value of around 8.0 was chosen for the experiments of Phase 1 and 2, according to the results obtained by Li et al., [29]. 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 μ m 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.

Table 1Concentrations of Ni and Cd in Phase 1 and Phase 2

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Heavy metals	Molar concentration (μ mol/L)
Ni	8.54
	38.87
	58.10
Cd	4.38
	21.90
	43.79

2.2 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 study of Rowe *et al.*, [30] structured a detailed recipe of synthetic landfill leachate that includes precise concentrations of organic matter (suspended and dissolved), ammoniacal nitrogen, phosphate and trace metals. The recipe was similar to a real leachate collected through a period of 3 months. The current study adapted the recipe of Rowe *et al.*, [30] with some modification of trace metals concentrations and NH₄-N content, as presented in Table 2. The same molar concentrations of Mg, NH₄ and PO₄, as well as concentrations of Ni and Cd, applied in Phase 1 were used during the preparation of synthetic landfill leachate in Phase 2.

Composition of synthetic landfill leachate (Adapted from Rowe *et al.*, [30])

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Component	Per 1 L
Acetic acid (100%)	5 mL
Propionic acid (100%)	1 mL
Butyric acid (100%)	1 mL
MgSO ₄ .7H ₂ O	17.253 g
Na ₂ CO ₃	0.120 g
Ca(NO ₃) ₂	0.100 g
NH ₄ Cl	3.744 g
NaH ₂ SO ₄	8.398 g

2.3 Analysis of samples

The concentration of NH₄-N in liquid samples was measured using Nessler's standard method, while PO₄ was measured using spectrophotometry analysis (HACH DR6000). Atomic adsorption

spectrophotometry (AAS, 3300, Perkin Elmer, USA) was used to analyse the concentrations of Ni and Cd in liquid samples. Solid samples of struvite precipitates were analysed using X-ray diffraction (XRD, Rigaku Americas Corporation, The Woodlands, TX) and Scanning Electron Microscopy (SEM, Gemini, Zeiss Supra Series, Germany).

3. Results

3.1 Removal of NH₄-N and PO₄ in the presence of Cd and Ni

Figure 1(a) – Figure 1(d) shows the removal percentage of NH₄-N and PO₄ when interacting with Cd and Ni. For Phase 1, as illustrated in Figure 1(a) and (c), the removal percentage for both NH₄-N and PO₄ was 92.56% and 97.74%, respectively. Meanwhile, for Phase 2, as illustrated in Figure 1(b) and (d), the removal percentages witnessed a minute change; removal of NH₄-N and PO₄ was 94.88% and 98.93%, respectively. The results are consistent with previous studies. Huang *et al.*, [31] achieved 92% removal of NH₄-N, and Miroslav *et al.*, [22] achieved 96% removal of PO₄ in wastewater by struvite crystallization.

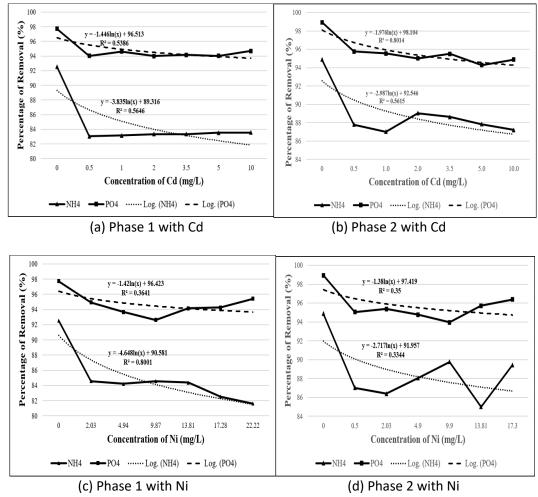


Fig. 1. Removal of NH₄-N and PO₄ in Phase 1 (a) & (c) and Phase 2 (b) & (d)

Referring to Figure 1 (a) and (b), based on the trendlines, the percentage of NH₄-N removal reduced as the initial Cd concentration increased. However, the reduction NH₄-N removal in Phase 1 was more significant than Phase 2. While for PO₄ removal, no significant changes were noticed since the curve of the plotted trendline fairly shows a straight line when the initial concentration of Cd

increases from 0.5mg/L to 10mg/L. Figure 1(c) and Figure 1(d) show the removal of NH₄-N and PO₄ when interact with Ni. Based on the trendlines, percentage of NH₄-N removal reduced as the initial Ni concentration increased. Similar to the interaction with Cd, there were no significant changes for the removal for PO₄. On the other hand, some fluctuations in NH₄-N removal were noticed (Figure 1 (d)), probably due to the interaction between the components in synthetic landfill leachate solution during the reaction. A study performed by Muhmood *et al.*, [32] shows that the removal percentage of Ni by struvite precipitation from poultry wastewater was 16.4% with significant amount of Ni in the final struvite precipitates.

3.2 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 pattern (Figure 2(a) and Figure 2(b)) and all the solid samples were in crystal form. However, the green pattern in the results displayed some background noise that could be due to amorphous compounds associated with the heavy metals ions of Ni²⁺ and Cd²⁺. Since the amorphous matter can be detected in XRD analysis, it can be said that the precipitates are in crystal form.

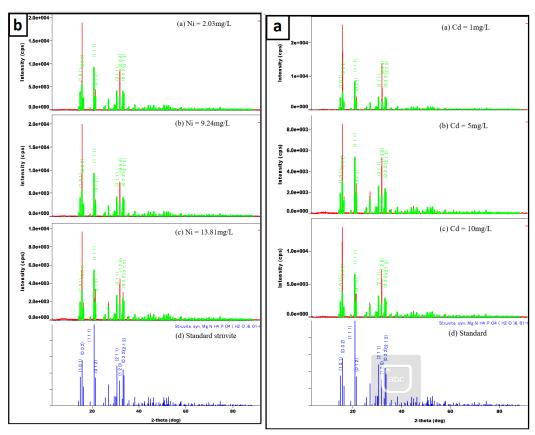


Fig. 2. XRD analysis of solid samples from Phase (1) in the presence of (a) Cd and (b) Ni

Struvite precipitates were further characterized by SEM analysis, for struvite obtained from the lowest and highest concentrations of Cd and Ni in Phase 1 and Phase 2, alongside with uncontaminated sample. Figure 3 – Figure 6 show the SEM images for the selected precipitates. In general, some differences were noticed between blanks obtained from Phase 1 and Phase 2; SEM images of blank from Phase 2 has obvious crested surface, numerous gullies, and the shape is more prismatic than the blank obtained from Phase 1. This might be due to the difference in the

composition of reactions solutions in both phases (i.e. synthetic solution and synthetic landfill leachate), in which the solution in Phase 2 contains organic acids that might have caused such difference. The observation is consistent with a previous study conducted by Tang *et al.*, [33], who used synthetic solution to produce struvite crystals.

Figure 3 and Figure 4 show the effect of Cd and Ni on struvite crystals' shape in Phase 1. It is illustrated that low Cd concentrations did not impose significant changes in the shape and surface of struvite crystals. Even at higher concentrations of Cd (10 mg/L), only a quite smoother surface was observed, which means that Cd did not affect the morphology of struvite when using the synthetic solution. This is consistent with the earlier results of spectrometry analysis of struvite contaminated with Cd. With regards to Ni, it showed clear holes on the surface of crystals, regardless of the low or high concentrations of Ni. This indicates that Ni has a more significant effect on the shape and morphology of struvite. On the opposite side, Cd and Ni in Phase 2 showed more obvious effect on struvite crystals. According to Figure 5 and Figure 6, X-shaped struvite crystals with blunt and round edges were formed (with Cd) and several holes were also noticed on the surface (with Ni), which was consistent with previous results obtained by Manzoor et al., [34] and Kemacheevakul et al., [35]. This means that the effect of Cd and Ni was slightly enhanced in the synthetic landfill leachate, probably due to an interaction with organic acids and other pollutants presented in the solution. Generally, based on the later discussion, struvite can be recovered safely in waste streams contaminated with Cd and Ni (within the examined ranges). This highlights the importance of conducting advanced researches in future to investigate the behavior and effect of heavy metals on struvite crystallization in actual waste streams.

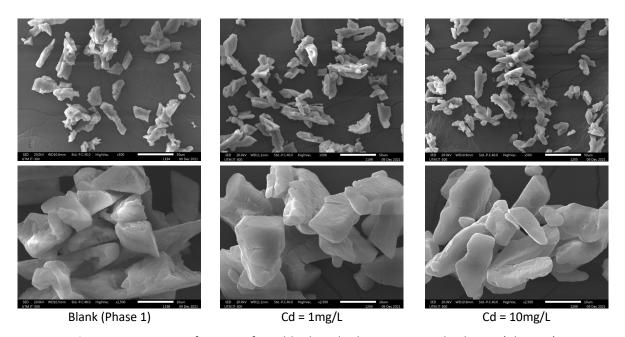


Fig. 3. SEM images of struvite from blank and Cd contaminated solution (Phase 1)

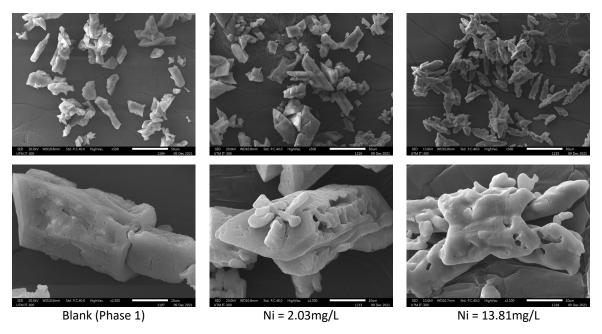


Fig. 4. SEM images of struvite from blank and Ni contaminated solution (Phase 1)

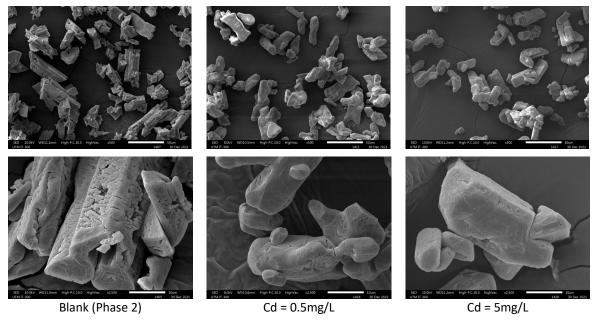


Fig. 5. SEM images of struvite from blank and Cd contaminated solution (Phase 2)

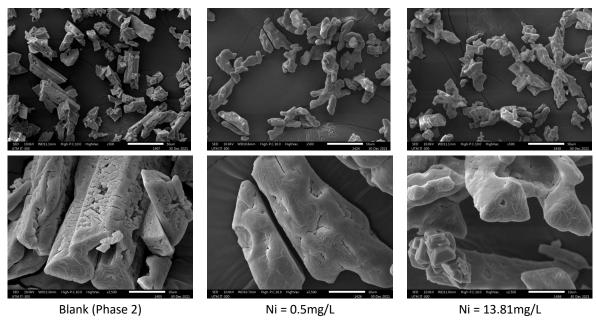


Fig. 6. SEM images of struvite from blank and Ni contaminated solution (Phase 2)

4. Conclusions

The recovery of NH₄-N from waste streams in the form of struvite is widely applied for several types of wastewaters, such as landfill leachate. However, heavy metals in landfill leachate could restrict the use of the obtained struvite, as it could attach to struvite crystals. The results of this study illustrated that Cd and Ni have minor effect on the purity and morphology of struvite, which was verified by AAS, XRD and SEM analyses. In conclusion, although the effect of Cd and Ni was insignificant, further studies have to be implemented to explore the effect of heavy metals on struvite in actual waste streams.

Acknowledgement

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