BEHAVIOR OF NICKEL AND CADMIUM IONS DURING STRUVITE CRYSTALLIZATION IN WASTEWATER

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ABSTRACT

The tremendous amounts of municipal solid wastes (MSW) generated contribute to the raise of nitrogen (N) in landfill leachate, which affect water bodies and cause severe damages to the environment. Struvite crystallization is a simple method that allows N recovery in the form of environmentally friendly fertilizer. Fundamentally, struvite ($MgNH_4PO_4.6H_2O$) is sensitive to changes in pH and the existence of foreign ions in the precipitation media. Therefore, heavy metals, such as nickel (Ni) and cadmium (Cd), in wastewater can reduce the quality of struvite. The current research studied the interaction behavior of Ni and Cd during struvite crystallization in landfill leachate. In addition, the thermal stability and microstructural composition under the equimolar ratio of struvite components was investigated. Struvite crystallization was conducted in synthetic solution (Phase 1) and synthetic landfill leachate (Phase 2) with pH maintained at 8.0±0.02. Struvite crystallization experiments proved that Cd and Ni have insignificant effect on the efficiency of PO_4 removal; however, they affect NH_4-N removal dramatically. The statistical analysis (ANOVA) shows that changing the experiment media from synthetic solution to synthetic landfill leachate did not affect the removal percentage of NH_4-N and PO_4. Atomic Absorption Spectrometry (AAS) analysis for liquid samples showed that the removal percentage of Ni and Cd was also high (65.68% - 99.35%), which proved that the destiny of the trace heavy metals was in solid samples. To further study the fate of Ni and Cd ions in struvite crystal, X-ray diffraction (XRD), scanning electron microscopy energy dispersive X-ray (SEM-EDX), and thermogravimetric analysis (TGA) analyses were conducted. The XRD results confirmed that highly pure crystals were formed. However, some crystals contained minor impurities such as trimagnesium phosphate, nitric acid dehydrate, nickel oxide and magnesium tetra-ammonium cyclotriphosphate tetrahydrate. In addition, SEM-EDX analysis showed that all struvite crystals interacting with Cd and Ni have changed in their surface structure, shape, and size. Moreover, the EDX analysis outcomes supported the results obtained from other analyses in Phase 1 and 2. TGA analysis for struvite showed that all struvite samples containing Cd and Ni in Phase 1 and Phase 2 were thermally stable until the thermal analysis ended with 43.32% - 45.45% remaining weight. The kinetics of struvite in the presence of Cd and Ni at pH 7.5 indicates that the kinetics constant increases up to 21.456 hr^{-1} with increasing Cd and Ni concentrations. The reaction of struvite with Cd at pH 8.5 followed the same trend. Meanwhile, the kinetics constants of struvite-Ni interaction at pH 8.5 demonstrate that the kinetics constant reduces to 6.408 hr^{-1}, which is lower than kinetics of pure struvite. In future, the outcomes in this study are expected to assist the struvite crystallization process in landfill leachate.

ABSTRAK

Jumlah sisa pepejal perbandaran (MSW) yang besar telah dihasilkan menyumbang kepada peningkatan nitrogen (N) dalam larut resapan tapak pelupusan, yang menjejaskan badan air dan menyebabkan kerosakan teruk kepada alam sekitar. Penghabluran *struvite* adalah kaedah mudah yang membolehkan pemulihan N dalam bentuk baja mesra alam. Pada asasnya, *struvite* ($MgNH_4PO_4.6H_2O$) adalah sensitif terhadap perubahan pH dan kewujudan ion asing dalam media pemendakan. Oleh itu, logam berat, seperti nikel (Ni) dan kadmium (Cd), dalam air sisa boleh mengurangkan kualiti *struvite*. Penyelidikan ini mengkaji tingkah laku interaksi Ni dan Cd semasa penghabluran *struvite* dalam larut resapan tapak pelupusan. Di samping itu, kestabilan terma dan komposisi mikrostruktur dalam nisbah molar yang sekata untuk setiap komponen *struvite* telah dikaji. Penghabluran *struvite* telah dijalankan dalam larutan sintetik (Fasa 1) dan larutan resapan tapak pelupusan sintetik (Fasa 2) dengan pH ditetapkan kepada 8.0±0.02. Eksperimen penghabluran *struvite* membuktikan bahawa Cd dan Ni mempunyai kesan yang tidak ketara ke atas kecekapan penyingkiran PO_4; walau bagaimanapun, ia mempengaruhi penyingkiran NH_4-N secara mendadak. Analisis statistik (ANOVA) menunjukkan bahawa menukar media eksperimen daripada larutan sintetik kepada larut resapan tapak pelupusan sintetik tidak menjejaskan peratusan penyingkiran NH_4-N dan PO_4. Analisis Spektrometri Serapan Atom (AAS) bagi sampel cecair menunjukkan peratusan penyingkiran Ni dan Cd juga tinggi (65.68% - 99.35%), yang membuktikan bahawa nasib logam berat surih adalah dalam sampel pepejal. Untuk mengkaji lebih lanjut nasib ion Ni dan Cd dalam kristal *struvite*, analisis pembelauan sinar-X (XRD), imbasan mikroskopi elektron, analisis sinar-X penyebaran tenaga (SEM-EDX), dan analisis termogravimetrik (TGA) telah dijalankan. Keputusan XRD mengesahkan kristal yang sangat tulen telah terbentuk. Walau bagaimanapun, sesetengah kristal mengandungi beberapa bendasing seperti *trimagnesium phosphate*, *nitric acid dehydrate*, *nickel oxide* and *magnesium tetra-ammonium cyclotriphosphate tetrahydrate*. Di samping itu, analisis SEM-EDX menunjukkan bahawa semua kristal *struvite* yang berinteraksi dengan Cd dan Ni menunjukkan perubahan dalam struktur permukaan, bentuk dan saiz. Lebih-lebih lagi, hasil analisis EDX juga menyokong keputusan daripada analisis lain dalam Fasa 1 dan 2. Analisis TGA untuk *struvite* menunjukkan bahawa semua sampel *struvite* yang mengandungi Cd dan Ni dalam Fasa 1 dan Fasa 2 adalah stabil secara termal sehingga analisis termal tamat dengan 43.32% - 45.45% baki berat. Kinetik *struvite* dengan kehadiran Cd dan Ni pada pH 7.5 menunjukkan bahawa pemalar kinetik meningkat kepada 21.456 hr^{-1} dengan peningkatan kepekatan Cd dan Ni. Tindak balas *struvite* dengan Cd pada pH 8.5 juga mengikuti trend yang sama. Sementara itu, pemalar kinetik untuk interaksi *struvite-Ni* pada pH 8.5 menunjukkan bahawa pemalar kinetik berkurang kepada 6.408 hr^{-1} yang mana lebih rendah dari pemalar kinetik untuk *struvite* tulen. Pada masa akan datang, hasil dalam kajian ini dijangka dapat membantu proses penghabluran *struvite* dalam larut resapan tapak pelupusan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AAS	-	Atomic Absorption Spectrophotometry
XRD	-	X-Ray Diffraction
SEM-EDX	-	Scanning Electron Microscopy-Energy Dispersive X-Ray
TGA	-	Thermogravimetric analysis
DTG	-	Differential Thermogravimetry
COD	-	Chemical Oxygen Demand
UTM	-	Universiti Teknologi Malaysia
N/A	-	Not Available
US	-	United States
PDF	-	Powder Diffraction File
MSW	-	Municipal Solid Waste
LL	-	Landfill Leachate
TOC	-	Total Organic Carbon
COD	-	Chemical Oxygen Demand

LIST OF SYMBOLS

L	-	Liter
mg	-	Miligram
mg/L	-	Miligram per liter
mL	-	Mililiter
µm	-	Micrometer
Å	-	Angstrom
∑	-	Sum
±	-	Plus-minus
R	-	Correlation Coefficient
h	-	Hour
hr^{-1}	-	Per Hour
min	-	Minute
min^{-1}	-	Per Minute
t	-	Time
C	-	Concentration
kg	-	Kilogram
%	-	Percent
Rpm	-	Rotation Per Minute
°C	-	Degree Celcius
cps	-	Count Per Second
deg	-	Degree
°C/min	-	Degree Celcius Per Minute
k	-	Kinetic Constant
Wt.	-	Weight

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Overview

Many types of wastewater are generated, including industrial, domestic, stormwater runoff, and landfill leachate. A high concentration of heavy metals and nitrogen in the wastewater urged the advancement of wastewater treatment technologies. This is because untreated nitrogen in wastewater will be discharged into the nearest water body and promote water pollution and other adverse environmental effects. The existing technologies focus on the efficiency of nitrogen removal, which is energy demanding. Instead, struvite emerged as one of the technologies that recover nitrogen efficiently and convert it into profitable end-products. Parallel with the sustainability policy of agriculture industries, the application of struvite as fertilizer is widely applied.

1.2 Problem Background

Since daily human activities depend on water, the quantity of wastewater generation has increased with the global population, industrialization and urbanization. Therefore, the composition of wastewater has been changing for many years. Types of wastewater are categorized based on the sources, i.e. different types of wastewater consist of different constituents. Major types of wastewater are industrial, domestic, stormwater runoff, and landfill leachate. Usually, wastewater comprises organic pollutants such as pathogenic microorganisms and inorganic pollutants such as nitrogen (N), phosphorus (P) and heavy metals, which are very harmful to the environment (Cervantes, 2009).

To date, approximately 57% of pollutants in the world are contributed by nitrogen (Nsenga Kumwimba *et al.*, 2018). Nitrogen in wastewater originates from protein, amines, composting plants, peat mines and peptides. Untreated nitrogen from wastewater will be conveyed into the nearest water body, causing water pollution. Some adverse effects are eutrophication and water acidification, which threaten the whole ecosystem. Thus, it is crucial to intensify the efforts to develop the best treatment technology to remove nitrogen from wastewater.

Nitrogen removal technologies have been thoroughly studied and implemented in many wastewater treatment plants worldwide. The technologies include biological, constructed wetlands, and chemical and physio-chemical processes. For biological processes, anaerobic ammonium oxidation (anammox) is an advanced technology that adopts the conventional nitrification/denitrification process. However, anammox technology removes nitrogen in wastewater and converts it into free nitrogen gas N_2 (Hu *et al.*, 2013).

Constructed wetlands are an effective nitrogen removal method (Camaño Silvestrini *et al.*, 2019). However, constructed wetlands have several disadvantages. The most obvious is the requirement for large land areas (Kivaisi, 2001). Physio-chemical processes such as ion exchange (Widiastuti *et al.*, 2008), air stripping (L. Zhang *et al.*, 2012), and adsorption (Halim *et al.*, 2010) are also widely. These technologies are highly efficient in terms of nitrogen removal; however, converting nitrogen into other forms of waste is the main drawback. Efforts for further treatment of removed nitrogen contribute to a significant increase in cost and energy consumption, which make the physio-chemical processes unsustainable.

The process of chemical precipitation of ammonium nitrogen (NH_4-N) forming magnesium ammonium phosphate (MAP) salt, or struvite, has been deeply studied since the 18th century. The crystallization process of struvite can be defined as a chemical process to remove ammonium nitrogen (NH_4-N) and phosphate (PO_4) in wastewater even at high ammonium nitrogen content in some wastewater, such as landfill leachate. Struvite chemical compound consist of magnesium (Mg^{2+}), ammonium nitrogen (NH_4^+) and phosphate (PO_4^{3-}) that shown in Equation (1.1).

$$Mg^{2+}{}_{(aq)} + NH_4^+{}_{(aq)} + PO_4^{3-}{}_{(aq)} + 6H_2O \leftrightarrow MgNH_4PO_4 . 6H_2O_{(s)} \quad (1.1)$$

Earlier, the scaling process of struvite was recognized as a problem in wastewater treatment plants. However, parallel with the high demand of nitrogen for agricultural activities, struvite emerged as a solution to recover ammonium nitrogen in wastewater. Mainly, struvite is used as an eco-friendly fertilizer, since it is found to be effective to plant and the roots due to the slow nutrient releasing property that avoids the roots burning (Rahman *et al.*, 2014). The application of struvite as fertilizer is widely investigated (Liu et al., 2013; Rahman et al., 2014; Ryu & Lee, 2016; Min et al., 2019).

Not only can it be applied as fertilizer, but struvite can also be developed as a fire-retardant barrier (Kaan *et al.*, 2018). Therefore, the major advantage of struvite is the capability to recover ammonium nitrogen into a valuable product through a simple and rapid process (Beckinghausen *et al.*, 2020).

However, heavy metals such as cadmium (Cd) and nickel (Ni) exist in the wastewater, especially landfill leachate, are becoming a threat to the purity of struvite formed and affecting the application of struvite as fertilizer where the concern for accumulation of heavy metals in plants arises. A recent study has shown that the plant’s capability to translocate Ni and Cd from landfill leachate into the plant tissue is very alarming (de Oliveira Mesquita *et al.*, 2021). Additionally, previous research reported that struvite is a potential adsorbent for Cd^{2+} ions (Wang *et al.*, 2017). Also, struvite tends to co-precipitate when Ni was introduced into the solution (Muhmood *et al.*, 2018) which reduce the efficiency of ammonium nitrogen recovery. Hence, a better understanding of Cd and Ni behaviour in wastewater is crucial to ensure the safety of food chain.

1.3 Problem Statement

Fundamentally, struvite is sensitive to changes in pH and the existence of foreign ions. Therefore, the presence of heavy metals such as nickel (Ni) and cadmium (Cd), as foreign ions, in landfill leachate can reduce the quality and purity of struvite, mainly when applied as a fertilizer and fire-retardant barrier material. The effect of some heavy metals on struvite crystallization, such as copper (Cu), zinc (Zn), chromium (Cr) and arsenic (As), has been widely studied and has shown some effects such as crack and fracture on the crystal' surface (Muryanto and Bayuseno, 2014) and reduce struvite kinetics constant (B. Li *et al.*, 2020). However, the effect of Ni and Cd was not investigated before, although they exist in landfill leachate.

As the interaction of Ni and Cd with struvite crystallization is still unknown, this research will study the interaction by focusing on the effect of thermodynamic stability and microstructural composition. Additionally, a kinetic study in different pH values has to be determined to understand the interaction of Ni and Cd with struvite. This research will focus on the interactions in landfill leachate by using synthetic landfill leachate. Plus, this research focuses on ammonium nitrogen instead of phosphate for nutrient recovery since landfill leachate contain higher amount of ammonium nitrogen compared to phosphate.

1.4 Research Objectives

The objectives of the research are:

(a) To investigate the fate of heavy metals Ni and Cd during struvite crystallization under equimolar ratio of struvite component.

(b) To discover the effect of Ni and Cd sorption on thermal stability and composition of struvite.

(c) To determine the kinetic of struvite crystallization in the presence of Ni and Cd under different pH value of solution.

1.5 Research hypothesis

Ni and Cd's fate or sorption mechanism during the struvite crystallization is expected to co-precipitate with struvite crystals instead of absorbed into the structure of struvite. The composition of struvite precipitate formed in the presence of Ni and Cd is also predicted to contain high content of Ni and Cd. Meanwhile, in terms of thermal stability, the effect of Ni and Cd sorption on struvite crystals is expected to increase the thermal stability, where the crystals with Ni and Cd start to decompose at a higher temperature than pure struvite. Lastly, the kinetics study of struvite also is expected to increase with the presence of Ni and Cd.

1.6 Scope of study

This study covers the behaviour of heavy metals cadmium (Cd) and nickel (Ni) during struvite precipitation in synthetic solution and synthetic landfill leachate to avoid interactions with other compounds and organic material contained in real wastewaters. The equimolar ratio (0.07M) of struvite components (Mg:N:P) is constant for all struvite crystallisation experiment. The same initial concentration of NH_4-N (990mg/L) were used in all experiments, which simulate the typical concentration of ammonium nitrogen in landfill leachate. Also, all experiments were batch crystallization experiment and conducted at room temperature and at a constant mixing speed of 40rpm with pH in range of 7.5 – 8.5. Meanwhile, the range of Cd and Ni was selected from lowest to highest typical concentration (0.5 – 22.2mg/L) in landfill leachate.

This research consists of three different phases (Phase 1, 2 and 3). Phase 1 and Phase 2 are the struvite crystallization in two different solution, synthetic solution and synthetic landfill leachate, respectively, with constant pH at 8.0. Phase 1 and 2 were designed to study the fate of Cd and Ni by measuring the initial and final concentration using AAS analysis, and their interactions with struvite in term of NH_4-N removal where the initial and final concentration of NH_4-N was analysed using spectrophotometric analysis (Nessler method). Meanwhile, the thermal stability of struvite was analysed using TGA analysis. Also, the composition of struvite was studied in Phase 1 and 2 where crystallinity of struvite samples were analyses using XRD analysis, and other characterization such as shape, surface's structure, and morphology were analysed using SEM-EDX analysis. Meanwhile, Phase 3 is designed to study the kinetics of struvite in the presence of Cd and Ni under different pH value of solution which are 7.5 and 8.5 where the initial and final concentration of Mg^{2+} is analysed using AAS analysis, and calculation of kinetics constant is done by using first-order kinetics model.

1.7 Limitation of study

This study focused on the recovery process of NH_4-N by struvite crystallization and how the behavior of Cd and Ni affected the struvite formed. However, there are some other aspect that were limited in this study, such as:

(a) The experiments in this study were performed in an equimolar ratio of struvite components, whereas the molar ratio is one of the main factors influencing struvite crystallization.

(b) The formation of struvite in this study was performed in synthetic solution and synthetic landfill leachate. This is because the formation of struvite is different in different wastewater.

1.8 Significant of study

Struvite crystallization technology is an effective and sustainable nitrogen removal and recovery process. Generally, this research will assist in mitigating water pollution by recovering nitrogen into potentially marketable products.

Besides that, this research will also provide a fundamental understanding of Ni and Cd interaction during struvite crystallization to access struvite purity. Hence, further contamination of heavy metals during struvite application could be avoided before being used in the agronomic field as an alternative fertilizer.

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