UNIVERSITI PUTRA MALAYSIA

MINIMIZATION OF NITROGEN-BASED CONTAMINANTS IN SURFACE WATER USING TREATED CLAY PARTICLES FOR PHYTOREMEDIATION ENHANCEMENT

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FK 2021 6

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By

NURUL SOLEHAH BINTI MOHD ZAINI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master Science

September 2020

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master Science

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September 2020

Chair : Mohd Nazli Naim, PhD
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Nitrogen contamination has become the main element of surface water pollution, leading to eutrophication and water quality deterioration that affect the aquatic ecosystem. The source of contaminant is majorly coming from the run-off fertilizer of the nearby agricultural activities. The long retention time of N contamination affects the surface water's ecosystem and green technology approaches such as phytoremediation come as a solution to water contamination issues. However, the phytoremediation process alone was less efficient in reducing the N contamination such as ammonium, NH_4^+ and nitrate, NO_3^- accumulation in the system and has caused the compound to accumulate in bulk instead of to be absorbed by the plant. Recent workers seek to improve the phytoremediation process as a safer treatment alternative to replace the commercial method that used physical and chemical treatment. Adsorption of the contaminants using clay particles serves as a green approach technique in polishing and improving the phytoremediation process. Therefore, the objectives of this study were to characterize the treated clay by adjusting the pH to acidic and basic condition followed by the spray dry method. Then, to analyze the adsorption capacity of the pH-treated clay particles of two different clay components; bentonite and kaolin, and to evaluate the enhanced phytoremediation process's water treatment performance using treated clays and macrophytes plant (*Nelumbo nucifera*). In this study, the clay particles were spray-dried after treated with varying pH conditions ranging from pH 2 to pH 10. The pH-treated clays were characterized by using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and X-Ray Fluorescence (XRF). The treated clay particles were subjected to the ammonium solution, and their adsorptive capacity was measured in terms of the ammonium removal efficiency, adsorption isotherm, and zeta potential value. The treated clay particles were subjected to the phytoremediation system, and the water treatment performance was evaluated. Nitrate was measured due to the conversion of ammonium to nitrate by microbes in the phytoremediation system. From the obtained results, the characterization results for SEM illustrated that bentonite’s surface has more

folded, richly wrinkled, and more pore compare to the less wrinkled and flat surface morphology of kaolin. XRF explained the presence of Fe and Ca element proving high isomorphous substitution in bentonite compare to kaolin. XRD showed the presence of montmorillonite mineral in bentonite that helps in the swelling capacity of bentonite. For the adsorption capacity, bentonite showed higher adsorption than kaolin with the maximum adsorption (q_{max}) of 7.463 mg/g and fitted well with the Langmuir model isotherm. Application of pH-treated clay particles obtained from the local soil sources with the *Nelumbo nucifera* plant successfully polished the water quality of the contaminated surface water by increasing the removal efficiency of NO_3^-> ninety-eight percent compared with the neutral pH-treated and control. The finding is expected to enhance the current phytoremediation practiced and help the local authorities to improve the quality of the contaminated surface water on a larger scale in the future.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PENGURANGAN BAHAN CEMAR BERASASKAN NITROGEN PADA PERMUKAAN AIR MENGGUNAKAN PARTIKEL TANAH LIAT YANG DIRAWAT UNTUK PENINGKATAN FITOPEMULIHAN

Oleh

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Pencemaran Nitrogen telah menjadi elemen utama kepada pencemaran permukaan air, menjurus kepada eutrofikasi dan kemerosotan kualiti air yang menjejaskan ekosistem akuatik. Sumber bahan cemar majoritinya datang dari pengaliran baja berdekatan dengan aktiviti pertanian. Pembendungan pencemaran N dalam tempoh masa yang lama mempengaruhi ekosistem permukaan air dan teknologi pendekatan hijau seperti fitopemulihan datang sebagai penyelesai kepada isu pencemaran air. Walaubagaimanapun, proses fitopemulihan secara sendirian kurang efektif dalam mengurangkan pencemaran N seperti pengumpulan ammonium, NH_4^+ dan nitrat, NO_3^- di dalam sistem dan telah menyebabkan kompaun terkumpul secara pukal daripada diserap ke dalam pokok. Pekerja baru-baru ini mencari cara untuk memperbaiki proses fitopemulihan sebagai rawatan alternatif yang selamat untuk menggantikan kaedah komersial yang menggunakan perawatan fizikal dan kimia. Penjerapan kompaun dengan menggunakan partikel tanah liat merupakan teknik pendekatan hijau dalam menggilap dan memperbaiki proses fitopemulihan. Oleh itu, objektif kajian ini ialah untuk mencirikan partikel yang telah dirawat dengan melaraskan pH kepada keadaan asid dan alkali diikuti dengan kaedah semburan kering. Kemudian, untuk menganalisis kapasiti penjerapan partikel pH-rawat tanah liat dari dua jenis komponen tanah liat; bentonit dan kaolin dan untuk menilai prestasi perawatan air proses fitopemulihan yang telah dipertingkatkan menggunakan tanah liat yang telah dirawat dan tumbuhan makrofit (*Nelumbo nucifera)*. Dalam kajian ini, tanah liat ini telah dikenakan semburan kering selepas dirawat dengan pelbagai kondisi pH bermula dari pH 2 hingga pH 10. pH-rawat tanah liat ini telah dicirikan dengan menggunakan Mikroskop Elektron Pengimbas, Difraksi Sinar-X dan Fluoresensi Sinar-X. Partikel tanah liat yang dirawat telah diuji ke atas larutan amonium dan kapasiti penjerapan diukur dari segi kecekapan penyingkiran amonium, isoterma penjerapan dan nilai potensi zeta. Partikel tanah liat yang dirawat telah diuji ke

atas sistem fitopemulihan, dan prestasi perawatan air telah dinilai. Nitrat telah diukur ekoran daripada penukaran amonium kepada nitrat oleh mikrob di dalam sistem fitopemulihan. Merujuk kepada keputusan yang diperolehi, keputusan pencirian untuk Mikroskop Elektron Pengimbas menunjukkan permukaan bentonit lebih berlipat, kaya dengan kedutan dan mempunyai lebih banyak pori berbanding dengan morfologi kaolin yang kurang kedutan dan lebih mempunyai prmukaan rata. Fluoresensi Sinar-X menerangkan kewujudan elemen ferum dan kalsium yang membuktikan penggantian isomorf yang lebih tinggi di dalam bentonit berbanding kaolin. Difraksi Sinar-X menunjukkan kewujudan mineral montmorilonit di dalam bentonit yang membantu kapasiti pembengkakan bentonit. Untuk kapasiti penjerapan, bentonit menunjukkan penjerapan yang lebih tinggi berbanding kaolin dengan penjerapan maksima sebanyak 7.463 mg/g dan mematuhi model isoterma Langmuir. Aplikasi pH-rawat tanah liat yang diperolehi daripada sumber tanah tempatan dengan pokok *Nelumbo nucifera* berjaya menggilap kualiti air permukaan air tercemar dengan meningkatkan kadar penyingkiran nitrat NO_3^-> sembilan puluh lapan peratus apabila dibandingkan dengan pH-rawat neutral dan kontrol. Pencarian diekspektasi mempercepatkan praktis fitopemulihan semasa dan membantu pihak yang bertanggungjawab untuk memperbaiki kualiti pencemaran permukaan air dalam skala yang besar pada masa hadapan.

ACKNOWLEDGEMENTS

By the name of Allah, The Most Gracious and The Most Merciful. Alhamdulillah, all praises to Allah S.W.T. for all the bestowed upon mankind and for giving me health and patience to accomplish this project.

My sincere appreciation goes to the Faculty of Engineering, UPM, and Department of Bio-Application and System Engineering, TUAT, Japan for the great concerns and opportunities given to accomplish this thesis project for the master course. I would like to express my deepest gratitude to my supervisor, Assoc. Prof. Ir. Dr. Mohd Nazli Naim and Prof. Wuled Lengoro for all the guidance given, useful advice, supervisions, concerns, constructive comments, and priceless assistance throughout this thesis project. It is an admiration to be under the supervision of an amazing person like them.

My sincere thanks also go to Assoc. Prof. Dr. Hasfalina Che Man, Dr. Siti Wahidah Puasa, and Assoc. Prof Dr. Noor Fitrah Abu Bakar as co-supervisors for this project, for their encouragement, insightful comments, guidance, and tolerance. Special thanks also go to Ms. Areej Amr Abdelazim Elkwiee and Mr. Norihiro Yoshida for all their cooperation and assistance throughout my project.

My sincere thanks to my parents, Mr. Mohd Zaini Bin Ismail and Mrs. Rapiah Binti Abdul Rafar, and family members for their love and continuous supports guide me to accomplish this project. Being accompanied by their love, I hold my strength to endure all the challenges faced in this project. Special appreciations to Ms. Tiara Nur Pratiwi, Mr. Mohd Syahlan Mohd Syukri, and Ms. Fatin Aina Zulkhairi Amin willing to spend their time and have supported me all the time throughout my project.

Besides, not forgetting Mr. Azhar Safwan, Ms. Nurul Ainina, Ms. Nur Syazwani, Ms. Heiza, and all my fellow friends, lab mates, and others whose I do not mention their names for helping me a lot without any vacillation. I will remember our sweet memories to make this thesis reality. Thanks for the bond of friendship and for making my life in UPM and TUAT full of untaught experiences and mesmerizing memories together.

Last but not least, I also owe a special thanks to Universiti Putra Malaysia for Graduate Research Funding (GRF), Ministry of Education Malaysia for Fundamental Research Grant Scheme (FRGS), and Japan Student Services Organization (JASSO) for monthly allowance in Japan.

I certify that a Thesis Examination Committee has met on 30 September 2020 to conduct the final examination of Nurul Solehah Binti Mohd Zaini on her thesis entitled “Minimization of Nitrogen Based Contaminants in Surface Water using Treated Clay Particles for Phytoremediation Enhancement” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

N	Nitrogen
NH_4^+	Ammonium
NO_3^-	Nitrate
SEM	Scanning electron microscopy
XRD	X-ray diffraction
XRF	X-ray fluorescence
WHO	World health organization
EPA	Environmental protection agency
AN	Ammoniacal nitrogen
WQI	Water quality index
INWQS	Interim national water quality standard
DO	Dissolved oxygen
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
SS	Suspended solids
DOE	Department of environment
NWQS	National water quality standard
Al^{3+}	Aluminium
Fe^{2+}/Fe^{3+}	Iron
Mg^{2+}	Magnesium
SiO_4^{2-}	Silicate
TOT	Tetrahedral:Octahedral:Tetrahedral
Si^{4+}	Silicon
Na^+	Sodium

K^+	Potassium
Ca^{2+}	Calcium
H^+	Hydrogen
OH^-	Hydroxide
CEC	Cation exchange capacity
Mn^{2+}	Manganese
Zn^{2+}	Zinc
Cu^{2+}	Copper
HSAB	Hard and soft acid base
IEP	Isoelectric point
HCl	Hydrochloric acid
NaOH	Sodium hydroxide
DLS	Dynamic light scattering
NH_4Cl	Ammonium chloride
Cl	Chloride
AlOH	Aluminium hydroxide
NRA	Nitrate reductase activity

CHAPTER 1

INTRODUCTION

1.1 Research Background

The human population has increased from 3.9 billion to 7.0 billion over the last decades, but the water demand has increased threefold. The world's need for water is growing twice as fast as the population (Lee et al., 2016). Malaysia, geographically located in the tropical region, receives an average rainfall of 3000 mm annually, which relatively rich in water resources (Abd Rasid et al., 2019; Lee et al., 2016). Water resources for human and ecosystem survival are derived primarily from the river, and 97 percent of the total water supply majorly comes from freshwater resources (Afroz et al., 2014; Othman et al., 2012).

However, rapid growth in urbanization, human population, and irrigated agriculture contribute to water contamination, i.e., run-off fertilizer into the water resources (Abd Rasid et al., 2019). Untreated contaminants from water run-off that inclusive of nitrogen-based contaminants enter surface water, i.e., rivers, lakes from which drinking water is drawn. These contaminants introduce a wide variety of toxic chemicals to the water used for drinking, cooking, and bathing (Zeliger, 2011). According to World Health Organization (WHO), maximum ammonium concentrations allowed in the surface water are 0.2 mg/L, 0.02 mg/L according to Environmental Protection Agency (EPA), and 1.5 mg/L according to European Legislation (Jeong et al., 2013). Conventional remediation approaches, which include a wide variety of physical, chemical, and thermal treatment alternatives or a combination of them such as ozonation, fat and grease removal, oxidation, and others to treat contaminated water would be too expensive (Champagne, 2007). In recent times, novel, cost-effective, and sustainable remediation strategies for removing or detoxifying contaminants are urgently needed as the major substitute for various chemical treating agents (Feng et al., 2017).

Phytoremediation provides a sustainable, economical, efficient, and versatile approach replacing the conventional treatment to improve the water quality (Abd Rasid et al., 2019; Champagne, 2007). Phytoremediation makes full use of the plant root system for the productive contaminants uptake (Jyoti, 2017). Lotus plant, also known as *Nelumbo nucifera* (*N. Nucifera*), is a herbaceous perennial aquatic plant used for phytoremediation (Jyoti, 2017; Kanabkaew & Puetpaiboon, 2004). The plant anatomy (leaves, stem, and rhizome) of N. Nucifera provides a good habitat for bacteria to attach and grow, contributing to a good phytoremediation system. N. Nucifera also has a unique gas transportation mechanism that can directly transport oxygen to the buried rhizome for microbial activities in degrading the contaminants that later will be uptake by the plants (Abd Rasid et al., 2019).

To enhance this phytoremediation system, clay particles have been introduced to increase the absorption or uptake of the contaminants by the plant root system (Champagne, 2007). The inclusion of clay particles close to the lotus plant roots increases the microbial activity (Champagne, 2007). Clay is the most promising material to solve this issue due to its superiority over commercially available adsorbents. The advantages of clay include abundant availability, low cost, non-toxicity, high adsorption properties, and large potential ion exchange (Vimonses et al., 2009). The utilization of clay as an adsorbent is also simple to operate, short contact time, and only requires energy from the sun (Malovanyy et al., 2013; M. K. Uddin, 2017). Clay particles such as bentonite consisted of montmorillonite's major component (Adeyemo et al., 2017; F. Uddin, 2018) and classified as 2:1 clay (Pecini & Avena, 2013). It has high adsorption capacity and swelling ability due to the interlayer space at the centre, causing the water and other molecules to enter and expand (Adeyemo et al., 2017; Kausar et al., 2018; Z. Xu et al., 2018). Another clay particle, such as kaolin, is mainly composed of kaolinite (Adeyemo et al., 2017; Vimonses et al., 2009), classified as 1:1 clay (Jinan Niu et al., 2019; F. Uddin, 2018). It is a non-swelling clay due to the absence of interlayer space (Au & Leong, 2013). The combination between the clay particles and plant is expected to enhance the plants' ability to perform its phytoremediation function to assimilate, concentrate and store the converted contaminants; i.e., inorganic N alternative to the commercial method that used physical and chemical processes.

1.2 Problem Statement

Malaysia received an average rainfall of 3000 mm and 566 billion water estimated running-off into the river system each year. This rainfall washed down the accumulated contaminants on land surfaces into the river system and affected the water quality (Afroz et al., 2014). According to Sim et al., more than 63% of Malaysia's rivers are classified as moderately to highly polluted. They are mostly polluted by agricultural runoff from fertilizer-rich lands such as vegetable farms, fruits and flower nurseries, golf courses, and animal farms (Sim et al., 2008).

The runoff contaminants that pollute the river include nitrogen-based contaminants, which are the main contributor to water eutrophication. This condition caused algal blooms, depleted dissolved oxygen in the water, and increased natural toxins in the water (Jeong et al., 2013; Zeliger, 2011). Ammonium accumulation, which can be further converted to nitrate and nitrite in nature, is toxic and dangerous to health when the substance's concentration exceeds 200 mg kg^{-1} body weight. The presence of ammonium in water also causes several effects, such as creating an odour in water, forming nitrite in the water supply system, impair manganese removal, and reducing disinfection efficiency (Mazloomi & Jalali, 2017).

Phytoremediation is more sustainable and low cost than the conventional approach getting more attention recently (Champagne, 2007). However, there

are some limitations to phytoremediation. According to Reddy and DeBusk, the storage of contaminants in the plants is short-term because of rapid turnover. The contaminants will be released back into the water if the plants are not harvested (Reddy & DeBusk, 1987). According to Ting et al., phytoremediation also faces the problem of longer retention times, ranging from 10 days to 3 years. Besides, phytoremediation is also not suitable for wastewater with extremely high Ammoniacal Nitrogen (AN) content (Ting et al., 2018). As a result, phytoremediation alone is a less efficient and time-consuming process (Kinidi & Salleh, 2017).

Previously, numerous studies have been done on the utilization of clay for the adsorption purpose (Adeyemo et al., 2017; Alshameri et al., 2018). The same goes for phytoremediation, and numerous studies have been done on an aquatic plant's ability to treat wastewater (Ting et al., 2018). However, the investigation of the soil used in the phytoremediation system is relatively scarce. This study helps to enhance the available phytoremediation process to provide better-treated wastewater. Therefore, to enhance the process of phytoremediation, clay particles that can attract the contaminants from the surrounding aqueous environment to its surface are introduced into the system (Adeyemo et al., 2017). The clay's porosity and high surface area caused strong physical and chemical interactions with the contaminants that contribute to the strong bonding power between nitrogen-based contaminants and the clay (M. K. Uddin, 2017).

1.3 Objective

The research's main objective was to study the effectiveness of clay in enhancing the phytoremediation process by using *Nelumbo nucifera*. The work can be divided into three parts:

1. To characterize the treated clay by adjusting the pH to acidic and basic condition followed by the spray dry method.
2. To analyze the adsorption capacity of the pH-treated clay particles of two different clay components; bentonite and kaolin.
3. To evaluate the enhanced phytoremediation process's water treatment performance using treated clays and macrophytes plant (Nelumbo nucifera).

1.4 Scope of Research

With the higher demand for green technology to treat wastewater, there has been a higher demand to improve the phytoremediation system. In view of this situation, this study analyzed pH-treated clay particles' use to enhance the phytoremediation system. To this end, the study covered the removal of nitrogen-based contaminants in an enhanced phytoremediation system. The study's scope is restricted to two different components of clay: bentonite and kaolin. The empirical study in this research is restricted to pH 2 to pH 10 treatment of clay particles. Further, the study also involved the characterization, zeta potential, and adsorption characteristics of clay particles. Therefore, the study's scope is

limited to the phytoremediation system, and more specifically, to utilize pH-treated clay particles in the phytoremediation system.

1.5 Thesis Structure

The first chapter discussed the study's background with the details of the study's aim, the problem statement, and the research scope. The second chapter provides a discussion based on the previous literature review that reflects the important findings in this study. Furthermore, this chapter also deliberates on the surface water pollution in Malaysia, especially focusing on nitrogen pollution. The phytoremediation process was discussed in terms of the improvements and challenges and the brief introduction of the clay application principles to enhance the phytoremediation process. The characterization analysis of the clay was reviewed to reveal the factor that affects the adsorption process. Chapter three provides an outline of the research methodology of this study. A detailed description of the adsorption and phytoremediation process's experimental set-up is used as the medium of research. This chapter highlighted several equations that were used to estimate the parameter needed for the study. Chapter four provides in-depth clay adsorptive capacity by studying the effect of ammonium adsorption principles using two different types of clays with various pH treatments to optimize the contaminant adsorption based on the particle's surface charge condition. The study also consists of the characterization of various pH-treated clays from a different group of clay. This chapter's discussion highlights the principles of clay adsorptive capacity for enhancing the phytoremediation process in treating the contaminated surface water. All obtained results were discussed fundamentally to relate the adsorption capacity with the N-cycle mechanism and chronology. This work is expected to understand the work principles between both relationships to enhance the phytoremediation process. Finally, chapter five concludes the findings and provides the modification of work for future recommendation.

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LIST OF PUBLICATIONS

Accepted

Zaini, N. S. M., Elkwiee, A. A. A., Naim, M. N., Bakar, N. F. A. Role of Nanoclay Surface Charge for Phytoremediation Process Enhancement. *Journal of Water Process Engineering*.

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