ADSORPTION OF Pb(II), Cu(II) AND Cd(II) FROM AQUEOUS SOLUTION BY NATURAL AND CHEMICALLY MODIFIED PINEAPPLE PLANT STEM

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By

VIVIAN LOH ZING TING

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

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

ADSORPTION OF Pb(II), Cu(II) AND Cd(II) FROM AQUEOUS SOLUTION BY NATURAL AND CHEMICALLY MODIFIED PINEAPPLE PLANT STEM

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August 2019

Chair : Tan Yen Ping, PhD
Faculty : Science

Pineapple plant stem (PPS) was used to sequester Pb(II), Cu(II) and Cd(II) from aqueous solution via adsorption. The PPS was chemically modified with organic acids and results showed the oxalic acid (OA) modified PPS (OAPPS) gave a better performance on the adsorption of Pb(II), Cu(II) and Cd(II) ions compared to natural PPS (NPPS). Both NPPS and OAPPS possessed hydroxyl, carbonyl and carboxyl functional moieties. The OAPPS has rougher surface and posed higher specific surface area than NPPS. NPPS and OAPPS have neutral surface charge at pH 4 and 2.4, respectively. The adsorption of Pb(II), Cu(II) and Cd(II) on NPPS and OAPPS from single metal solution showed similar trends. The removal percentages of Pb(II), Cu(II) and Cd(II) reached equilibrium at 0.05 g in 25mL adsorbate solution and optimum adsorption were noted at pH 4 – 6. The adsorption capacities of NPPS and OAPPS for Pb(II), Cu(II) and Cd(II) increased with the adsorbate concentration and reached maximum capacities at the equilibrium time of 30 – 90 min. The experimental data were found to best fitted with pseudo-second order kinetics model and Langmuir isotherm model. The maximum monolayer adsorption capacities of NPPS were 14.09, 5.08, and 1.50 mg/g while OAPPS achieved at 30.96, 8.54, and 3.64 mg/g for Pb(II), Cu(II), and Cd(II), respectively. The increase of temperature lowered the adsorption capacities of NPPS and OAPPS for Cu(II) and Cd(II) ions but enhanced the Pb(II) adsorption, suggested that Pb(II) adsorption is endothermic while Cu(II) and Cd(II) adsorption are exothermic reactions in nature. The interference ions in aqueous solution reduced the adsorption capacities of NPPS and OAPPS for Cu(II). Surprisingly, these co-existing ions enhanced the adsorption performance of NPPS for Pb(II) and Cd(II) in aqueous solution, however, it did not happen the same on OAPPS. The acid medium was found to be a better eluent for the regeneration of exhausted adsorbents and NPPS and OAPPS could be reused for 3 consecutive cycles.

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The surface complexation, electrostatic attraction, physisorption and chemisorption were elucidated for the adsorption of Pb(II), Cu(II) and Cd(II) on NPPS and OAPPS. The adsorption of Pb(II), Cu(II) and Cd(II) from aqueous solution using NPPS and OAPPS were carried in binary and ternary metal system as well. The results showed that the effect of binary and ternary metal system on the adsorption capacity of NPPS and OAPPS was antagonistic as the adsorption capacity of both adsorbents decreased when the system changed from single to binary or ternary. The NPPS and OAPPS adsorbed the metal ions in the order of Pb(II) > Cu(II) > Cd(II). Finally, NPPS and OAPPS were applied in real wastewater from textile industry and both adsorbents showed their ability to adsorb Pb(II) and Cd(II) from the wastewater. In general, the results proposed that PPS has more room for improvement and could be a potential adsorbent for Pb(II), Cu(II) and Cd(II) elimination from aqueous solution.

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

PENJERAPAN Pb(II), Cu(II) DAN Cd(II) DARI LARUTAN AKUEUS MENGGUNAKAN BATANG TUMBUHAN NANAS YANG SEMULAJADI DAN YANG DIMODIFIKASI DENGAN BAHAN KIMIA

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Batang pokok nanas (PPS) telah digunakan sebagai penjerap untuk mengurangkan Pb(II), Cu(II) dan Cd(II) dari larutan akueus melalui penjerapan. Data menunjukkan PPS yang dimodifikasikan dengan asid oksalik (OAPPS) memberikan prestasi yang terbaik dalam penjerapan ion-ion Pb(II), Cu(II) dan Cd(II) berbanding dengan PPS yang semulajadi (NPPS). NPPS dan OAPPS mempunyai kumpulan berfungsi seperti hidroksil, karbonil dan karboksil. OAPPS mempunyai permukaan yang lebih kasar dan kawasan permukaan yang lebih tinggi daripda NPPS. NPPS dan OAPPS mempunyai caj permukaan neutral pada pH 4 dan 2.4 masing-masing. Penjerapan Pb(II), Cu(II) dan Cd(II) atas NPPS dan OAPPS menunjukkan trend yang sama. Peratusan penyingkiran Pb(II), Cu(II) dan Cd(II) mencapai keseimbangan pada 0.05 g dalam 25 mL larutan akueus dan penjerapan optimum diperhatikan pada pH 4 – 6. Kapasiti penjerap NPPS dan OAPPS untuk Pb(II), Cu(II) dan Cd(II) meningkat dengan kepekatan ion-ion logam dan mencapai keseimbangan pada masa 30 – 90 min. Data eksperimen berpadanan dengan model kinetik tertib pseudo-kedua dan model Langmuir untuk penjerapan Pb(II), Cu(II) dan Cd(II). Kapasiti monolayer maksima untuk NPPS ialah 14.09, 5.08, dan 1.50 mg/g manakala OAPPS mencapai 30.96, 8.54, dan 3.64 mg/g untuk Pb(II), Cu(II), dan Cd(II). Peningkatan suhu menurunkan penjerapan Cu(II) dan Cd(II) tetapi meningkatkan penjerapan Pb(II) dari larutan akueus, menunjukkan bahawa penjerapan Pb(II) adalah endotermik manakala penjerapan Cu(II) dan Cd(II) adalah tindak balas eksotermik. Kemunculan ion-ion berdampingan dalam larutan akueus telah menurunkan kapasiti penjerapan Cu(II) pada NPPS dan OAPPS. Sebaliknya, ion-ion berdampingan ini meningkatkan prestasi penjerap NPPS untuk Pb(II) dan Cd(II), namun, hal ini tidak berlaku pada OAPPS. Medium asid didapati eluan yang lebih baik untuk regenerasi penjerap, NPPS dan OAPPS boleh diguna balik sebanyak 3 kali.

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Kompleks permukaan, daya tarikan elektrostatik, penjerapan fizikal dan kimia telah dijelaskan untuk penjerapan Pb(II), Cu(II) dan Cd(II) atas NPPS dan OAPPS. Penjerapan Pb(II), Cu(II) dan Cd(II) dari larutan akueus menggunakan NPPS dan OAPPS juga dikaji dalam sistem penduaan dan pertigaan. Data eksperimen menunjukkan bahawa kapasiti penjerapan NPPS dan OAPPS adalah bersifat antagonistik dalam kedua-dua sistem tersebut kerana kapasiti penjerapan NPPS dan OAPPS menurun apabila sistem tunggal berubah ke sistem penduaan atau pertigaan. NPPS dan OAPPS menjerap dalam susunan Pb(II) > Cu(II) > Cd(II). Akhirnya, NPPS dan OAPPS digunakan dalam air kumbahan dari industri tekstil dan berupaya menjerap Pb(II) dan Cd(II). Hasil kajian mencadangkan bahawa PPS mempunyai lebih banyak ruang untuk penambah baikan dan berpotensi menjadi penjerap untuk mengurangkan ion-ion Pb(II), Cu(II) dan Cd(II) dari larutan akueus.

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This is to confirm that:

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Signature: ______________________________
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Signature: ______________________________
Name of Member of Supervisory Committee: Assoc. Prof. Dr. Abdul Halim Abdullah

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LIST OF ABBREVIATIONS

PPS	Pineapple plant stem
NPPS	Natural pineapple plant stem
CAPPS	Citric acid modified pineapple plant stem
OAPPS	Oxalic acid pineapple plant stem
BET	Brunauer-Emmett-Teller
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared
PZC	Point of zero charge
q_e	Amount of ions adsorbed at equilibrium
q_t	Amount of ions adsorbed at time
C_i	Concentration of ion before adsorption
C_f	Concentration of ion after adsorption
K	Rate constant
k_1	Rate constant of pseudo-first order kinetics model
k_2	Rate constant of pseudo-second kinetics model
R^2	Correlation coefficients
q_{max}	Maximum adsorption capacity
b	Langmuir constant related to energy of adsorption
K_f	Frendlich constant relating to adsorption capacity
n	Freundlich isotherm exponent constant
$\Delta G°$	Free energy of adsorption
$\Delta H°$	Enthalpy of adsorption
$\Delta S°$	Entropy of adsorption
R	Gas constant
1/T	Reciprocal of temperature

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LIST OF APPENDICES

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CHAPTER 1

INTRODUCTION

1.1 Research background

Water is the greatest resource, as the surface of Earth is largely covered. However, only 0.7% of fresh water is available and can be used by living organisms (National Geographic Society, 2012), henceforth the importance of water is irreplaceable. Industrial activities are inevitable as it is crucial for maintaining the economic stability and living sustainability. Large scale industrial use of heavy metals, such as lead (Pb), copper (Cu), and cadmium (Cd) caused these hazardous heavy metal ions are widespread in the water environment through industrial effluents. The concentration of heavy metal ions is particularly high in areas of mining, metal processing refineries, paper processing and textile facilities. Heavy metal ions are bio-accumulative in the ecosystem, and can be transferred through food chain or drinking water to the human body. While some trace metal ions like Cu(II) are essential micro-nutrients to human, others such as Pb(II) and Cd(II) are harmful to human even in tiny quantities. Water pollution with heavy metal ions is a global concern owing to its damaging effect to the human body and the nature environment. Therefore, industrial effluent should be treated to permissible concentration threshold before discharging into the water stream.

Water purification and separation technologies have gained tremendous attention since they play a vital role in regulating the amount of heavy metal ions released from industrial effluent. The removal of heavy metal ions from water has achieved by methods such as ion exchange (Rao *et al.*, 2005), membrane filtration (Francis *et al*., 2016), photocatalysis (Kanakaraju & Wong, 2018), electrochemical treatment (Tran *et al*., 2017), chemical precipitation and coagulation (Pang *et al*., 2009). Adsorption is an emerging technique as it promotes low operating costs, minimizing the volume of waste to be disposed of, and good efficiency in removing metal ions from solution (Kumar, 2006). Activated carbon is a common adsorbent for adsorption but has high production cost. The search for low cost and easily available adsorbents has led to the investigation on agricultural waste materials as potential adsorbents of metal ions.

Agricultural wastes are included as lignocellulosic plant materials, mainly consist of cellulose, hemicellulose, and lignin. A number of works have proposed to reuse plant waste materials as adsorbents due to the high availability and economic friendliness. Thus, in this research, adsorption capacity of a typical lignocellulosic plant biomass namely pineapple (*Ananas comosus*) plant stem is considered for the removal of Pb(II), Cu(II) and Cd(II) from aqueous solution. The untreated plant biomass have been observed to

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give lower adsorption capacity (Wang *et al*., 2014) and leaching of soluble organic compounds from the plant biomass (Šćiban *et al*., 2007; Azadeh *et al*., 2015) caused secondary water pollution with higher chemical oxygen demand (COD), biological oxygen demand (BOD) and total organic carbon (TOC) in the water environment. Pre-treatments and chemical modifications on plant materials are reported to solve the problems (Rabemanolontsoa & Saka, 2016) as organic matters were removed, functional groups were exposed and additional binding sites were embedded in the cell wall of biomass, thereby enhanced the adsorption performance of plant waste materials (Garba *et al*., 2016).

This study aims at assessing the adsorption potential of the adsorbents prepared from the natural and chemically modified pineapple plant stem (PPS) for the removal of Pb(II), Cu(II) and Cd(II) from aqueous solution. The interactive effects of process parameters such as the adsorbent dosage, solution pH, initial metal ion concentrations, contact time, temperature and the presence of co-existing ions on the adsorption capacity of adsorbents towards the three metal ions were studied in single metal aqueous solution. Desorption studies were carried out to determine the best eluent for the removal of Pb(II), Cu(II) and Cd(II) from the surface of adsorbents, and regeneration studies on the prepared adsorbents were also conducted to test the reusable potential of PPS. Comparisons were made between the natural and chemically modified PPS on their physical and chemical characteristics as well as the adsorption capacities towards Pb(II), Cu(II) and Cd(II) in single metal aqueous solution. Metal ions adsorption mechanism was also suggested by the kinetics, isotherm and thermodynamics studies. Besides, adsorption capacity of PPS was evaluated in binary and ternary metal systems to study its metal ion selectivity. Finally, the capabilities of natural and chemically modified PPS have also been tested in real wastewater.

1.2 Problem statements

Heavy metals such as lead (Pb), copper (Cu), and cadmium (Cd) are widely involved in manufacturing, pharmaceutical, batteries, metal refining, and textile industries. These industrial activities release untreated effluent and contaminate the water environment with heavy metal ions. High concentration of Pb(II) and Cd(II) was reported by Muhammd (2018) from tannery and textile effluents in Ethiopia, East Africa. Besides, the amount of Cu(II), Pb(II) and Cd(II) was reported high in India from sugar industry, steel processing, pharmaceutical, paints, textile, and battery industries effluents, exceeded permissible concentration limits (PCL) set by EPA standards (Suresh *et al*., 2015; Silambarasan *et al.*, 2012). Heavy metal ions are toxic due to their non-biodegradable, long half-life properties, and bio-accumulative in human body, thus damages organs especially the liver and kidney. The quality of water around the globe is lowered and this rise as global environmental issue. Therefore, it is necessary to reduce the concentration of dangerous metal ions in the water sources.

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Harmful heavy metal pollutants released from industrial wastewater give negative effects to the human body, as well as the environment. Unfortunately, current wastewater treatment methods have shortcomings, namely high capital and maintenance costs, along with generation of secondary hazardous pollutants. The existing wastewater treatment method was observed insufficient in removing most metal ions from industrial effluent. The concentration of Pb(II) and Cd(II) in treated textile effluent was remained high and greater than the PCL (Fenta, 2014). Therefore, research was conducted to suggest, find and develop economically feasible, simple to operate, efficient and sustainable method so as to protect the surface water bodies.

Agricultural activities are active all around the world and wastes are largely produced, this event then created disposal problems. Pineapple plant is a commercial fruit crop, and its fruit is commonly involved in food industry. The generation of solid by-products such as pineapple crown, stem, peels and pulp is a vexing society problem. Open burning is one of the methods to dispose these agro-wastes and this activity gives rise to air pollution. Hence, in hopes to combat the agricultural waste disposal problem, pineapple plant stem (PPS) is reused as an adsorbent for the current adsorption method.

The use of lignocellulosic plant materials without any pre-treatments and modifications is not suitable to be used as adsorbents as it leached organic pollutants. The organic matter in water will give rise to environmental problem such as eutrophication, lowered COD and BOD level in water. The accumulation of nutrients in water stimulates the growth of algae as a result the surface of water was covered, oxygen level in water depleted, and the aquatic living organisms were negatively affected. Hence, an appropriate routine to prepare agro-waste adsorbents is necessary to counter the leaching of organic pollutants to the aquatic system.

This work focused on the elimination of Pb(II), Cu(II) and Cd(II) from single metal aqueous solution using agro-waste, pineapple plant stem (PPS) as adsorbent. The preparation of the adsorbents was conducted by simple and cost affordable method. The heavy metal ions adsorption performance on PPS was then investigated under different operation conditions.

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1.3 Objectives

The general objective of this project is to utilize the abundant and locally available agricultural waste materials, pineapple (*Ananas comosus*) plant stem for heavy metal ions removal from aqueous solution.

The specific objectives of this project include:

i. To synthesize and characterize the natural and chemically modified pineapple plant stem (PPS) as adsorbent.

ii. To study the batch adsorption of Pb(II), Cu(II) and Cd(II) by using natural and chemically modified PPS under various variables such as adsorbent dosage, solution pH, initial metal ion concentration, contact time, and temperature in aqueous solution.

iii. To examine the characteristics of kinetics, isotherms, and thermodynamics for the adsorption of Pb(II), Cu(II) and Cd(II) on PPS.

iv. To evaluate the reusable potential of natural and chemically modified PPS by desorption and regeneration studies.

1.4 Scope of thesis

There are five chapters in this thesis and the following is the scope of each chapter:

Chapter 1 provides the research background, problem statements, and the objectives of this study.

Chapter 2 presents a literature review of the uses and hazardous effects of Pb(II), Cu(II) and Cd(II) ions, the conventional wastewater treatment techniques, the adsorbents used in heavy metal ion adsorption, and chemical modifications on lignocellulosic plant materials. A brief introduction on the pineapple uses, wastes, and its application on the removal of metal ions is included in Chapter 2 as well.

The methods for preparing the natural pineapple plant stem (NPPS) and chemically modified pineapple plant stem are described in Chapter 3. The experimental conditions of the batch adsorption studies are also described.

Chapter 4 examines the effectiveness of pineapple plant stem (PPS) to remove Pb(II), Cu(II) and Cd(II) from aqueous solution by adsorption. The mathematical modeling of kinetics, isotherms, and thermodynamics on the adsorption of Pb(II), Cu(II) and Cd(II) by NPPS and oxalic acid modified pineapple plant stem

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(OAPPS) have been decribed in this chapter. Chapter 4 also included the desorption and regeneration studies of NPPS and OAPPS, the possible mechanisms on the Pb(II), Cu(II) and Cd(II) adsorption by NPPS and OAPPS, and comparison of NPPS and OAPPS adsorption capacities. The studies on selectivity and competitiveness of Pb(II), Cu(II) and Cd(II) on developed adsorbents, application of NPPS and OAPPS for Pb(II), Cu(II) and Cd(II) removal from real wastewater are described in Chapter 4 as well.

General findings from this work and several suggestions for future research are summarized in Chapter 5.

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