UNIVERSITI PUTRA MALAYSIA

ADSORPTION OF COPPER(II) AND LEAD(II) IONS BY PALM KERNEL SHELL-DERIVED ACTIVATED CARBON

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ADSORPTION OF COPPER(II) AND LEAD(II) IONS BY PALM KERNEL SHELL-DERIVED ACTIVATED CARBON

By

EKEMINI MONDAY ISOKISE

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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DEDICATION

To my caring parent, Dr. and Mrs. M. A. Ntok

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 COPPER(II) AND LEAD(II) IONS BY PALM KERNEL SHELL-DERIVED ACTIVATED CARBON

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July 2019

Chairman : Abdul Halim bin Abdullah, PhD
Faculty : Institute of Advanced Technology

In the wastewater treatment plant, activated carbon is a widely used adsorbent to remove heavy metals and organic pollutants, but it is very expensive. Therefore, adsorption by utilizing different types of agro-residues is one of the alternative materials to remove various contaminant from solutions. Due to the high toxicity of Pb and Cu as trace metal pollutants in the environment, many studies have dedicated to suggest possible ways of eliminating these metals from the environment. This study focus on preparation of activated carbon from Palm Kernel Shell as economically and environmental friendly adsorbent for removal of Pb^{2+} and Cu^{2+} from aqueous solution. The activated carbon prepared were mainly mesoporous in nature with BET surface area and isoelectric point (IEP) ranged from 1004 to 1083 m^2/g and 2.8 to 3.1, respectively. Effect of operating parameters such as activated carbon dosage, contact time, temperature, metal ion concentration and pH were investigated. Adsorption capacity was found to vary with initial concentration, adsorbent dose and pH. An increase in pH led to a significant increase in heavy metal removal suggesting the involvement of ion exchange mechanism. Adsorption kinetics, isotherms and thermodynamics parameters of the metal ions sorption process were also evaluated. Pseudo-second-order kinetics explained the adsorption process satisfactorily, which suggests chemisorption as the rate limiting step and mechanism for the removal of Cu^{2+} and Pb^{2+}. The Langmuir isotherm model was most suitable for describing the adsorption process. The monolayer saturated adsorption capacities of AC-600 2:1(4) for Pb^{2+} and Cu^{2+} was 114.9 mg/g and 27.93 mg/g, respectively. Therefore, the prepared palm kernel shell based activated carbon found to be efficient in removing heavy metal.

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

PENJERAPAN TEMBAGA (II) DAN PLUMBUM (II) ION MENGGUNAKAN KARBON TERAKTIF TERBITAN KELOMPANG KELAPA SAWIT

Oleh

EKEMINI MONDAY ISOKISE

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Dalam loji rawatan air kumbahan, karbon teraktif telah digunakan secara meluas sebagai penjerap untuk menyingkirkan logam berat dan bahan pencemar organik, tetapi kosnya adalah sangat mahal. Oleh itu, penjerapan dengan menggunakan pelbagai jenis sisa-tani adalah salah satu bahan alternatif untuk menyingkirkan pelbagai larutan bahan pencemar. Disebabkan oleh tahap keracunan tinggi Pb dan Cu sebagai bahan pencemar logam alam sekitar, banyak kajian telah mencadangkan langkah-langkah untuk menyingkirkan logam-logam ini dari alam sekitar. Kajian ini tertumpu kepada penyediaan karbon teraktif berasaskan tempurung kelapa sawit sebagai penjerap yang menjimatkan dan mesra alam untuk menyingkirkan Pb^{2+} dan Cu^{2+} dari larutan akueus. Karbon teraktif yang disediakan bersifat mesoporous mempunyai kawasan permukaan BET dan titik isoelektrik (IEP) masing-masing antara 1004 hingga 1083 m^2/g dan 2.8 hingga 3.1. Kesan operasi parameter seperti dos karbon teraktif, masa sentuhan, suhu, kepekatan awal logam ion dan pH telah dikaji. Kapasiti penjerapan didapati berbeza dengan kepekatan awal, dos penjerap dan pH. Peningkatan pH membawa kepada peningkatan ketara penyingkiran logam berat yang menunjukkan penglibatan mekanisme pertukaran ion. Penjerapan kinetik, isoterma dan parameter termodinamik bagi proses erapan logam ion turut dinilai. Kinetik pseudo-aturan-kedua menjelaskan proses penjerapan dengan memuaskan, ini menunjukkan bahawa penjerapan kimia sebagai mekanisme yang berkemungkinan dalam menyingkirkan Cu^{2+} dan Pb^{2+}. Model Isoterma Langmuir adalah paling sesuai dalam menjelaskan proses penjerapan. Kapasiti penjerapan ekalapisan tepu AC-600 2:1 (4) bagi Pb^{2+} dan Cu^{2+} adalah masing-masing adalah 114.9 mg/g dan 27.93 mg/g. Kesimpulannya, tempurung, kelompang kelapa sawit yang berasaskan karbon teraktif didapati berkesan sebagai penyingkir logam berat.

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Ekemini Monday Isokise, March 2019

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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Declaration by graduate student

I hereby confirm that:

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectrometer
AC	Activated carbon
AC-600 2:1 (1)	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 2:1 for 1 hour activation time
AC-600 2:1 (2)	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 2:1 for 2 hours activation time
AC-600 2:1 (3)	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 2:1 for 3 hours activation time
AC-600 2:1 (4)	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 2:1 for 4 hours activation time
AC-PKS	Palm kernel shell activated carbon
BJH	Barrett-Joyner-Halenda
BET	Brunauer-Emmet-Teller
IEP	Isoelectric point
PKS	Palm kernel shell
pH_{pzc}	Point of zero charge
UNICEF	United Nations International Children’s Emergency Fund
WHO	World Health Organization

CHAPTER 1

INTRODUCTION

1.1 Background Studies

Metals, including copper (Cu), lead (Pb), Zinc(Zn) etc., present in water bodies are not degradable and bound to cause serious environmental damages due to their toxicity. In the meantime, the removal of heavy metals from aqueous waste has received increasing attention in the last decade. Due to the high toxicity of Pb and Cu as trace metal pollutants in the environment, many studies have dedicated on their interaction with adsorbents (Pyrgaki et al., 2018). The respective allowable concentration of Pb and Cu ions in drinking and wastewater are 0.01 mg/L and 0.05 mg/L, and 0.002 mg/L and 0.005 to 30 mg/L, according to World Health Organization benchmark (WHO, 2008). Adsorption is one the methods that is commonly used in the removal of these heavy metals (Zuo, 2014) because of the advantages; including simple operation, low cost, and low secondary environmental pollution and the ability to remove heavy metals at less than 100 mg/L when other methods cannot work effectively (Tang et al., 2017). Varieties of adsorbents, such as activated carbon, resin, chitosan, have been so far developed. Particularly, activated carbon has been widely used to remove heavy metals from various wastewaters based on the larger surface area and porosity. Recently, attention has been shifted to the use of waste materials in the preparation of activated carbon due to the high cost associated with the use of coal and lignite as precursors. Because of the abundance of palm kernel shell (PKS), the recovery of value-added products is greatly desirable (Xu et al., 2017). Thus, the research and application of PKS' rudimentary performance have been given great attention since it shows to be a promising precursor for the production of AC (Xu et al., 2017).

The activating agents are important in tailoring the textural properties in PKS activated carbons. Among $ZnCl_2$, KOH, NaOH and ortho phosphoric acid (H_3PO_4), H_3PO_4 is the most widely used activating agent (Kumar and Jena, 2016). These chemicals are dehydrating in nature, and influence the pyrolytic decomposition and prevent the formation of tar (Sayğılı and Güzel, 2018).

Using H_3PO_4, numerous studies have been carried out so far on the preparation of ACs and they have focused on the influence of concentration of the impregnation solution and soaking temperature on the porous structure (Sivachidambaram et al., 2017). However, little or no attention has been given to the influence of retention time on the pore structure of the activated carbon. Thus, is of interest to consider the matter. Previous results agree that the activation of biomass by phosphoric acid in a ratio of 2:1 at temperature of 600 ^{o}C, produced ACs of highest BET surface area (1559.9 m^2/g), pore size (1.71 nm) and total pore volume (0.303 cm^3/g) (Pam et al., 2018).

Considering this literature, this study aimed to examine the effects of retention time on the porous structure and adsorption capacity of AC produced from palm kernel shell using phosphoric acid in a ratio of 2:1(acid: precursor wt/wt) at temperature of 600 ^{o}C. The products were characterized in terms of adsorption of Pb(II) and other laboratory analyses (surface area analysis (BET)) to evaluate their physical and chemical properties.

1.2 Problem Statement and Justification

In order to minimize the risk of pollution caused by heavy metals on the environment, this research was conducted to study the adsorption characteristics of lead and copper from aqueous solution using activated carbon. The use of methods, such as electrochemical, ion exchange, chemical precipitation have some limitations, including low efficiency, generation of secondary pollution, and high cost of operation. Besides, these methods require high level of expertise, consequently they are not applied by lots of end-users. Adsorption by adsorbent is promising especially with activated carbon. However, commercial activated carbon is expensive. Hence, there is growing interest in finding adsorbents that are cost effective with fewer limitations. In this work, activated carbon was prepared from palm kernel shell and used to treat Cu and Pb from aqueous solution.

1.3 Significance of the Research

The findings will be useful in the development of low cost technology in the removal of toxic heavy metals from contaminated water using agricultural by-products as adsorbents. In addition, the use of palm kernel shell as adsorbents will undoubtedly serve as an economic solid waste management strategy. These will contribute to the sustainability of the surrounding environment.

1.4 Aim and Objectives of Research

The main aim of this study is to prepare activated carbon from PKS using H_3PO_4 with following specific objective.

Specific Objectives

(i) To prepare and characterize activated carbons from palm kernel shells
(ii) To determine the adsorption capacity of the activated carbon under different parameters of contact time, operational temperature, initial ion concentration and pH in removing the heavy metals from wastewater.
(iii) To evaluate the adsorption kinetics, adsorption isotherms and thermodynamics parameters of the adsorption process.

1.5 Scope of work

The scope of this work include the following:

(i) Preparation of palm kernel shell activated carbons (AC-PKS)
(ii) Characterization of the AC-PKS.
(iii) Screening of AC-PKS for the removal of heavy metal ion (Pb(II))
(iv) Batch adsorption studies – effect of different parameters in adsorption process
(v) Kinetics, isotherms and thermodynamics evaluation of the adsorption process

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BIODATA OF STUDENT

Ekemini Monday Isokise is a Nigerian born on the 15th day of December, 1981. He is the first son and second child in a family of five children to Mr and Mrs Monday Archibong Ntok. He has bagged numerous degrees from different highly recognised universities. These include Bachelor's degree in Geography and Regional planning from the University of Calabar, Nigeria. He later obtained his Post Graduate Diploma (PGD) degree in Environmental Resource Management from the University of Uyo, Nigeria. His latest educational achievement is the completion of a Master's degree in Green Engineering from the Universiti Putra Malaysia. Prior to bagging his PGD and also the Master's degree, Ekemini has had some experience in the educational sector during his one-year compulsory National service, where he served as a Geography teacher at Tofi Memorial Grammar School, Gboko Local Government Area of Benue State, Nigeria. Despite bagging these degrees, suffice here to say that Ekemini Isokise is far from reaching his limit in educational achievements, but rather, has just begun.

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