UNIVERSITI PUTRA MALAYSIA

REMOVAL OF Ni (II) AND Pb (II) IONS FROM AQUEOUS SOLUTION USING AMBERLYST-15 ION EXCHANGE RESIN AS AN ADSORBENT

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By

NAYMA MOHAMED ASALMI ALAMIN

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

May 2017

DEDICATION

I would like to dedicate my work to my family members and both my parents for their full support to carry out my Master Degree Study in Universiti Putra Malaysia

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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Chairman : Abdul Halim Bin Abdullah, PhD
Faculty : Science

Heavy metals are among the most inorganic hazardous pollutants. Therefore, the removal of metal ions in man-made and natural water sources is essential in order to protect the various ecosystems.

Adsorption is by far the most versatile technique for removal of metal ions especially when adsorbent regeneration and reuse could improve cost effectiveness.The high cost and difficulty of separating of activated carbon restrict its application in wastewater treatment. Organic ion exchange resins are found more suitable for the removal of toxic elements due to their faster kinetics, ease of regeneration and high exchange capacity. Thus the present study was aimed to evaluate the potential use of Amb-15 ion exchange resin as a an adsorbent for Ni(II) and Pb(II) metal ions. To achieve this aim, the adsorption experiments were performed to investigate the efficiency of Ni(II) and Pb(II) removal in wide variety of experimental parameters .

The adsorption of metal ions onto the adsorbent was found to be highly dependent on the adsorbent dose, the initial concentration of metal ions and contact time. Solution pH showed an uneven positive effect on the metal ions due to the different mechanism dominate the adsorption interactions (ion exchange and chemisorption).

Thermodynamics, kinetics and adsorption equilibrium parameters studies suggest, the importance of chemical reaction between Am-15 and the metal ions.

The maximum adsorption efficiencies and capacities obtained from the single batch system were 93.3 and 99.9 % and 112.5 and 384.6 mg g^{-1} for Ni(II) and Pb(II) respectively. Which sufficiently good compared to several ion exchange resin or synthesized adsorbents derived from natural materials.

In conclusion, the bath adsorption procedure was applied on Sungai Balok Kuantan Pahang river water sample and up to 80% and 92% removal were obtained for Ni(II) and,Pb(II) respectively. Overall, ion exchanger Am-15 commercially available can be effectively used as an adsorbent for heavy metal removal from the synthetic and natural water sample.

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

PENYINGKIRAN ION Ni (II) DAN Pb (II) DARI AIR LARUTAN AQUEOUS MENGGUNAKAN RESIN PENUKARAN AMBERLYST-15 SEBAGAI PENJERAP

Oleh

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Logam berat adalah antara bahan pencemar berbahaya paling tidak organik. Oleh itu, penyingkiran ion logam dalam sumber buatan manusia dan sumber semula jadi adalah penting untuk melindungi pelbagai ekosistem.

Adsorpsi adalah teknik yang paling serba boleh bagi penghapusan ion logam terutamanya apabila regenerasi dan penggunaan semula penyerap dapat meningkatkan keberkesanan kos. Kos yang tinggi dan kesukaran memisahkan karbon diaktifkan membatasinya dalam rawatan air sisa. Resin pertukaran ion organik didapati lebih sesuai untuk penghapusan unsur-unsur toksik akibat pergerakan mereka yang lebih cepat ,memudahkan penjanaan semula dan kapasiti pertukaran tinggi. Oleh itu, kajian ini bertujuan untuk menilai potensi penggunaan resin pertukaran ion Am-15 sebagai penyerap untuk penyingkiran ion Ni(II) dan Pb(II). Untuk mencapai matlamat ini, eksperimen penjerapan dilakukan untuk menyiasat kecekapan pengasingan Ni(II) dan Pb(II) dalam pelbagai parameter eksperimen.

Penjerapan ion logam ke dalam penjerap didapati sangat bergantung kepada dos penjerap, kepekatan awal ion logam dan masa kenalan. Penyelesaian pH menunjukkan kesan positif yang tidak sekata terhadap ion logam kerana mekanisme yang berbeza menguasai interaksi penjerapan (pertukaran ion dan chemisorption).

Kajian parameter thermodynamics, kinetics dan adsorption mencadangkan, kepentingan reaksi kimia antara Am-15 dan ion logam.

Kecekapan dan kapasiti penjerapan maksimum yang diperoleh daripada sistem tunggal batch adalah 93.3 dan 99.9% dan 112.5 dan 384.6 mg g-1 untuk Ni(II) dan Pb(II). Masing-masing adalah cukup baik berbanding resin pertukaran beberapa ion atau penjerap sintetik yang diperoleh daripada bahan semula jadi.

Kesimpulannya, prosedur penjerapan batch telah digunakan pada sampel sungai seperti Sungai Balok Kuantan Pahang dan sehingga 80% dan penyingkiran 92% diperolehi untuk Ni(II) dan Pb(II). Secara keseluruhan, penukar ion Am-15 yang boleh didapati secara komersil boleh digunakan secara berkesan sebagai penjerap untuk pengalihan logam berat dari sampel air sintetik dan semula jadi.

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

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LIST OF ABBREVIATIONS

AM-15	Amberlyst-15
AC	Activated Carbon
AAS	Atomic Absorption Spectroscopy
BET	Brunauer, Emmet and Teller Isotherm Model
IPD	intra-particle diffusion model
FESEM	Filed Scanning Electron Microscopy
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometer
USEPA	United States Environmental Protection Agency
WHO	World Health Organization
UF	Ultrafiltration
NF	Nanofiltration
RO	Reverse osmosis
CO	Chemical oxidation

LIST OF SYMBOLS

C_e	Equilibrium metal ions concentration (mgL^{-1})
C_0	Initial metal ions concentration in solution (mgL^{-1})
C_t	Concentration of metal ions in solution at time t (mgL^{-1})
R_L	Separation factor of Langmuir
T	Temperature (K)
k_1	Rate constant of pseudo-first-order kinetic model (min^{-1})
k_2	Rate constant of pseudo-second-order kinetic model ($g\ mg^{-1}\ min^{-1}$)
M	Mass of adsorbent used (g)
R^2	Correlation coefficient value
1/n	Adsorption intensity (heterogeneity)
Q_t	Amount of heavy metal ions adsorbed at time t, mgg^{-1}
Q_{max}	Maximum adsorption capacity for complete monolayer ($mg\ g^{-1}$)
F	Fractional attainment of equilibrium at time t
n	Freundlich constant related to adsorption intensity
K_F	Adsorption capacity at unit concentration ($mg\ g^{-1}$) $(Lmg^{-1})^{1/n}$
k_C	Thermodynamic adsorption equilibrium constant
ΔG^0	Gibb's free energy of adsorption ($kJmol^{-1}$)
ΔH^0	Change in enthalpy of adsorption ($kJmol^{-1}$)
ΔS^o	Entropy change of adsorption ($kJmol^{-1}$)
V	Volume of adsorbate (L)

M	Mass of adsorbent used (g)
Q_d	Amount of metal ions desorbed (mg g^{-1})
C_d	Concentration of metal ions after desorption (mg L^{-1})
R	Percentage of metal ions removal
$C_{boundary}$	Boundary layer thickness
k_{id}	Intra-particle diffusion rate constant (mg g^{-1} $min^{-0.5}$)
b	Langmuir constant related to adsorbent-metal binding affinity
Ri	Initial adsorption value
R	Universal gas constant, 8.314 J $mol^{-1}.K^{-1}$

CHAPTER 1

INTRODUCTION

1.1 Introduction

Water is finite, scarce and of vital significance in our everyday lives and therefore the imperative to enhance and safeguard the quality of water is extremely important. Point and non-point sources of water pollution are quickly sullying our valuable water resources. The pointed types of water pollutants may be classified as inorganic, organic and biological in nature. The most widely recognised inorganic water toxins are heavy metals, which are profoundly harmful and cancer-causing in nature. In addition to nitrates, the sulphates, phosphates, fluorides, chlorides and oxalates are also having serious impacts. Pesticides which include a variety of compounds are the main sources of organic pollutants found in wastewater (Gupta *et al*., 2012).

The main source of heavy metal contamination found in the environment is industrial wastewater. This includes effluents resulting from electroplating, metal finishing and from within printed circuit board industries including the manufacturing of batteries. These industries produce annually, a significant amount of wastewater containing heavy metal ions (Wong *et al.,* 2013).

1.2 Problem statement

Although small quantities of some heavy metals are essential for human health and well-being, an excess amount of these metals can have a negative and harmful impact on our health (Chowdhury *et al*., 2016). Heavy metal contamination likewise, can adversely affect the development of living organisms, thus weakening and potentially altering the dynamics in our human society, which in turn may potentially impact our future generation's ability to survive (Au *et al*., 2001). Hence, indiscriminate disposal of wastewater is causing environmental concerns globally given the number of negative consequences on human health (Wang and Chen , 2006).

Due to their unique physical and chemical properties,Pb(II) and Ni(II) can be used in broader manufacturing applications as within the battery, glassware and painting industries and are commonly present in industrial effluents (Bailey *et al.*, 1999; Abdel-Wahab *et al*., 2015; Bhatnagar and Minocha, 2010). Pb(II) has been defined as being a priority hazardous substance having harmful toxic effects and it is widely documented as causing anaemia, diseases of the liver and kidneys, brain damage and even death (Jain *et al*., 1989). Ni(II) also poses a threat to living organisms due to its immunological and genetic toxicity, carcinogenicity and genetic mutations. Since Pb (II) and Ni (II) are both classified as being the most hazardous of the heavy metals in the chemical-intensive industries, these toxic heavy metals must be removed from wastewater to prevent their release into the environment (Barakat, 2011; Chung *et al*., 2014; Ngah and Hanafiah 2008; Yu *et al.*, 2000).

Removal of heavy metal ions from inorganic effluent can be achieved by conventional treatments such as through chemical precipitation or flotation. However, these techniques have considerable disadvantages, given that the corrosive-resistant equipment is quite overpriced, the need for energy is high and the difficulty to implement these techniques in developing countries. Adsorption has been shown to be an economically feasible alternative method for removing heavy metals from wastewater (Allaire-Leung et *al*., 2000; Babel and Kurniawan, 2003a).

The ideal adsorbent to remove heavy metal ions is assumed to possess the following characteristics: a large surface area, suitable total pore volume and average pore diameter, high adsorption capacity and selectivity, mechanically stable, being easily available and the possibility of regeneration and being environmentally safe (Markovic *et al*., 2015; Rafatullah *et al*., 2010).

Various materials have been used to eliminate heavy metal ions from aqueous solutions including the use of clay (Abollino *et al.*, 2003), palm shell activated carbon (Onundi *et al.*, 2010), Meranti sawdust (Rafatullah *et al.*, 2009), peat (Bartczak *et al.*, 2015), olive stone (Martín-Lara *et al*., 2014) and chitosan (Sargn and Arslan, 2015). Among these adsorbents, activated carbon is regarded as one of the most effective adsorbents used in heavy metal ion removal due to its high specific surface area and adsorption capacity. However, it is relatively expensive and difficult to separate thereby restricting its use and therefore, fungible and effective adsorbents are required for heavy metal contaminated water (Zhang *et al.*, 2016).

Ion exchange resins are normally exploited to remove heavy metal either as sulfonic acid cation exchangers in hydrogen form or quaternary ammonium and ion exchangers in hydroxide form. It is worth noting that these resins are insoluble in the reaction medium and can be easily removed by simple filtration and therefore, steps and equipment are required for adsorbent removal to be reduced. Furthermore, they can be handled easily and safely (Lakouraj *et al.*, 2006). Ion exchange resins such as Amberlite IRC-718, Purolite1 S930 and Dowex have been reported to have a high ion exchange capacity for Ni(II) and Pb(II) and able to treat wastewater to an appropriate quality in compliance with regulations in force (Stefan and Meghea, 2014).

In the present study, Am-15 cation exchange resin was used to remove Ni(II) and Pb(II) ions from an aqueous solution. The ability of the Am-15 resin to remove Ni(II) and other metal ions (iron and chromium) using ion exchange method has been reported (Shaaran, 2009) , and noted that the resin has also been employed to absorb chromium(III) from aqueous solutions .The resin revealed fast kinetics and high exchange capacity towards Cr(III) (Mustafa *et al*., 2010).

However, Involving of Am-15 resin use to remove Pb(II) is not available in the literature. Besides using the resin via adsorption process to adsorb the Ni (II) ions is not available as well. Thus, it is of great interest to study the adsorption characteristics of Ni (II) and Pb (II) onto Am-15.

The adsorption performance of the resin was discussed in terms of the effects of the adsorbent dose, the initial heavy metal ions concentration, pH solution and temperature. The possibility of the repeated use of the resin in the removal of both heavy metal ions has been studied through desorption-adsorption experiments.

1.3 Research Objectives

The main aim of this work is to investigate the adsorption properties of Am-15 resin towards Ni (II) and Pb (II) metal ions in aqueous solution. Thus the specific objectives are listed as below.

1. To determine the efficiency and capacity of the adsorbent for Pb (II)and Ni (II) ions uptake using several parameters such as adsorbent mass, contact time, solution temperature and initial concentration and pH.
2. To characterize the adsorption process using kinetic study, adsorption isotherms and film and intra-particle diffusion model.
3. To evaluate the reusability efficiency of Am -15 in removing Pb (II) and Ni(II) from aqueous solution.

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