REMOVAL OF LEAD FROM AQUEOUS SOLUTION BY GREEN SYNTHESIS OF ADSORBENTS USING SAPODILLA LEAVES EXTRACT

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DEDICATION

To all the people who gave me hands through the journey.

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ABSTRACT

Rapid development of population, industrialization, and urbanization leads to global contaminants of water pollution by heavy metal ions that cause threat to the health of human and other living creatures. Out of all heavy metal pollutants, lead should be taken into consideration due to it detrimental effects. Among all available treatment methods for this issue, adsorption has been found to be better in terms of simplicity of design, cost effective, ease of operation, and environmentally friendly. Currently, activated carbon is one of the most used and popular commercial adsorbent for treating of heavy metal from aquatic medium. However, its application is limited due to relatively high commercial cost. The use of low cost biomass waste to produce adsorbents has been shown to provide economical solution to this problem. This study aims to develop waste based adsorbents using sapodilla (*Manilkara Zapota)* leaves extract along with Fe salt as coating materials for alumina beads (Fe/C/Al_2O_3), commercial molecular sieve (Fe/C/M.S), and cockle shell (*Anadara Granosa*) (Fe/C/C.S). All of the adsorbents were prepared through wetness impregnation method. The adsorbents were tested trough batch adsorption studies with several operating conditions such as initial metal ion concentration, adsorbent dosage, and contact time in order to evaluate their effect on adsorption process. The Atomic Adsorption Spectrophotometer (AAS) was then applied to determine the remaining of metal ions in sample solution. The results revealed that the removal efficiency of Pb(II) ions is the highest for Fe/C/C.S adsorbent at optimum condition (60 mg L^{-1} metal ion concentration, 1.5 g dosage, and 4 h of contact time). The equilibrium modelling showed that the sorption of Pb(II) was better fitted by Langmuir model for Fe/C/Al_2O_3 and Fe/C/M.S, while Freundlich model for Fe/C/C.S. Furthermore, the kinetic of adsorption of all adsorbents for Pb(II) better described by pseudo-second order kinetic model. The reusability testing of potential adsorbent (Fe/C/C.S) revealed that adsorbent are capable to employ up to four cycles. Meanwhile, the stability of adsorbent investigated using X-ray fluorescence (XRF) showed that adsorbent was stable. Furthermore, X-ray diffraction (XRD) study revealed the polycrystalline structure of adsorbents. Analysis trough nitrogen adsorption (NA) and field emission scanning electron microscopy (FESEM) showed that adsorbents are mesoporous with rough and uneven surface. Energy dispersive X-ray (EDX) analysis confirmed the presence of Al, C, O, Fe, Si, Ca elements in the adsorbents. Fourier transform infrared spectroscopy (FTIR) analysis revealed the presence of several functional groups on the adsorbents. Due to its removal efficiency, reusability, and stability, Fe/C/C.S adsorbent can be considered to be an economically advantageous material for treating Pb(II) ions from aqueous solution.

ABSTRAK

Pembangunan pesat penduduk, perindustrian, dan perbandaran membawa kepada pencemaran global pada air oleh ion logam berat yang menyebabkan ancaman kepada kesihatan manusia dan makhluk hidup lain. Daripada semua logam berat pencemar, plumbum perlu dipertimbangkan kerana ia memberi kesan buruk. Di antara semua rawatan yang ada untuk masalah ini, penjerapan telah didapati lebih baik dari segi kesederhanaan reka bentuk, kos efektif, kemudahan operasi, dan mesra alam sekitar. Pada masa ini, karbon aktif adalah salah satu penjerap komersil yang paling banyak digunakan dan popular untuk merawat logam berat dari medium akuatik. Tetapi, penggunaannya terhad kerana kos komersial yang agak tinggi. Penggunaan sisa biomass kos rendah untuk menghasilkan penjerap telah menunjukkan dapat memberikan penyelesaian ekonomi kepada masalah ini. Kajian ini bertujuan untuk menghasilkan penjerap berasaskan sisa menggunakan ekstrak daun sapodilla (*Manilkara Zapota*) bersamaan dengan garam Fe sebagai bahan pelapisan untuk manik alumina ($Fe/C/Al_2O_3$),penapis molekular (Fe/C/M.S) dan kulit kerang (*Anadara Granosa*) (Fe/C/C.S). Semua penjerap disediakan melalui kaedah pengisitepuan basah. Penjerap diuji melalui kajian penjerapan batch dengan beberapa keadaan operasi seperti kepekatan ion logam, dos penjerap, dan masa hubungan untuk menilai kesannya terhadap proses penjerapan. Spektrofotometer serapan atom (AAS) kemudian digunakan untuk menentukan sisa ion logam dalam larutan sampel. Hasilnya menunjukkan bahawa kecekapan penyingkiran ion Pb(II) adalah tertinggi untuk penjerap Fe/C/C.S pada keadaan optimum (kepekatan ion logam 60 mg L^{-1}, dos 1.5 g, dan masa 4 jam). Pemodelan keseimbangan menunjukkan bahawa penjerapan Pb(II) lebih melengkapi model Langmuir untuk $Fe/C/Al_2O_3$ dan Fe/C/M.S, dan model Freundlich untuk Fe/C/C.S. Selain itu, kinetik penjerapan semua penjerap untuk Pb(II) lebih diterangkan oleh model kinetik tertib pseudo-kedua. Ujian kebolehgunaan semula dari penjerap yang berpotensi (Fe/C/C.S) menunjukkan bahawa penjerap mampu digunakan sehingga tiga kitaran. Sementara itu, kestabilan penjerap yang disiasat menggunakan pendarfluor sinar-X (XRF) menunjukkan bahawa penjerap adalah stabil. Tambahan pula, kajian difraksi sinar-X (XRD) menunjukkan struktur polikristalin penjerap. Analisis melalui nitrogen adsorpsi (NA) dan mikroskopi elektron pengimbas pemancaran medan (FESEM) menunjukkan bahawa penjerap mesoporous dengan permukaan kasar dan tidak sekata. Analisis spektroskopi serakan tenaga sinar-X (EDX) mengesahkan kehadiran elemen Al, C, O, Fe, Si, Ca dalam penjerap. Spektrometer pengubah fourier inframerah (FTIR) menunjukkan kehadiran beberapa kumpulan berfungsi pada penjerap. Disebabkan oleh kecekapan penyingkiran, kebolehgunaan semula, dan kestabilan, penjerap Fe/C/C.S dapat dianggap sebagai bahan yang menguntungkan dari segi ekonomi untuk merawat ion Pb (II) daripada larutan akueus.

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LIST OF ABBREVIATIONS

WHO	-	World Health Organization
DOE	-	Department of Environment Malaysia
FESEM	-	Field Emission Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
NA	-	Nitrogen Adsorption
FTIR	-	Fourier Transform Infrared
XRF	-	X-Ray Fluorescence
MS	-	Molecular sieve
CS	-	Cockle Shell
SLE	-	Sapodilla Leaves Extract
BET	-	Brunnauer, Emmet and Teller
IUPAC	-	International Union of Pure and Applied Chemistry
BJH	-	Barret-Joyner-Halenda

LIST OF SYMBOLS

q_e	-	Adsorption Capacity
R^2	-	Correlation Coefficient
q_{max}	-	Maximum Adsorption Capacity
n	-	Adsorption Capacity Constant
K_f	-	Freundlich Constant
K_L	-	Langmuir Constant
K_1	-	Pseudo First-Order Constant
K_2	-	Pseudo Second-Order Constant
γ	-	Wavelength
λ	-	Gamma
θ	-	Half Angle of Diffraction Beam

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

It is a fact that the urbanization leads to massive escalation in industrialization that involves machines, and agricultural activities. One of products of this trend is increase the water pollution level, especially heavy metal contamination (Gogoi et al., 2018). Heavy metals are the group of trace elements such as metals and metalloids with an atomic density greater than 4 ± 1 g cm^{-3} (Nagajyoti et al., 2010). The most common heavy metals found as pollutants are ions such as cadmium, lead, zinc, nickel, copper, mercury and chromium or their compounds (Renu et al., 2017). All of these materials are difficult to degrade and can be accumulated in food chain. Due to it continuous present in food chain, it can cause serious health problem in human body (Chen et al., 2018).

Among all of heavy metal contaminants, lead poses crucial concern. Lead can cause central nervous system damage. Also, lead can render several problems such as damaging the kidney, liver and reproductive system, basic cellular processes and brain functions (Fu and Wang, 2011). Common symptoms of lead poisoning are confusion, headache, abdominal pain, anemia, irritability and coma (Mittal et al., 2016). Therefore, it is necessary to tackle down this issue by discovering methods to reduce heavy metal contaminants level in waste water.

Numerous methods have been used to overcome heavy metal contaminants problem such as chemical precipitation, membrane separation, electrochemical treatment, coagulation-flocculation, ion-exchange, and adsorption. However, adsorption is considered to be one of the most affordable and effective method (Burakov et al., 2018). Adsorption involves mass transferring process in which a substance is carried to solid surface from a liquid phase, and interacts chemically and/or physically (Carolin et al., 2017). Physical forces involve van der waals force (dipole moments, polarization forces, dispersive forces, or short range repulsive interactions) while chemical forces are valency forces arising out of the redistribution of electrons between the solid surface and the adsorbed atoms (Bansal et al., 2005). Moreover, all adsorption methods are reliant on solid-liquid equilibrium and on mass transfer rates (Kegl et al., 2020).

In this research, the application of alumina beads (Al_2O_3), molecular sieve (M.S), and cockle shell (C.S) adsorbents are mainly focused for adsorbing lead (Pb(II)) from aqueous solution. Sapodilla leaves extract and iron oxide (Fe_2O_3) were used as coating for alumina (Fe/C/Al_2O_3), molecular sieve (Fe/C/M.S), and cockle shell (Fe/C/C.S). Sapodilla leaves extract was used as source of carbon (C) which acts as binder between Fe and support material as well as source of functional groups for enhancing adsorption capacity. Fe is a potential material as adsorbent since it is abundantly available on earth, environmentally friendly, and high removal capacity for heavy metal ions (Hua et al., 2012).

Meanwhile, cockle shells were used as calcium carbonate ($CaCO_3$) sources. The use of $CaCO_3$ as adsorbent materials is gained attention because of their omnipresence in nature and high sorption affinity to most heavy metals (Du et al., 2012). Extraction from dried sapodilla leaves and waste cockle shells are used because of their wide availability and as an attempt to reduce the waste problem. In line with this, common conventional adsorbents, commercial molecular sieve (Al_2O_3/SiO_2), and alumina beads (Al_2O_3) is an adequate choice for adsorbents due to their non-toxicity, inexpensiveness, high adsorption capacity, and environmentally friendly.

Sapodilla (*Manilkara zapota L.*), family: Sapotaceae (Figure1.1 (a)) is orginated in Mexico and central America which can be found in most tropical areas all around the world like Central and South America, the West Indies, India, Florida in the United States, Sri Lanka, Indonesia and Malaysia. Sapodilla possess various name such as Ciku, Chickle gum, Chiku, Chikoo and Naseberry (Khaliq et al., 2019).

Despite of its tasteful fruit that contains sugar, protein, minerals, sapodilla leaves have also been applied as traditional medicine for curing diarrhea, cough, cold and pulmonary infections (Ling and Esa, 2019;Kishore and Mahanti, 2016).

Furthermore, Cockles (*Anadara Granosa*), as displayed in Figure 1.1 (b), are a kind of shellfish that dominate on muddy areas which act as affordable protein sources in South East Asian region. In Malaysia, cockles market is profitable businesses which reached over RM 91.60 million profit in 2010 (Muthusamy et al., 2016). Moreover, during 2010 cockles were produced about 10 tonnes yearly in Selangor alone (Mohamed, Yousuf, et al., 2012). In line with this, in 2011 to 2017 the production of cockles in Malaysia was up to 8024.70 tonnes annually (Saffian et al., 2020). Hence, the waste of cockles shell can be serious issue if the shells are dumped without any proper treatment. Cockles shell mainly consist of calcium carbonate ($CaCO_3$) that have potential in environmental remediation such as biosorbent for removal heavy metal ions from aqueous solution (Rashidi et al., 2011).

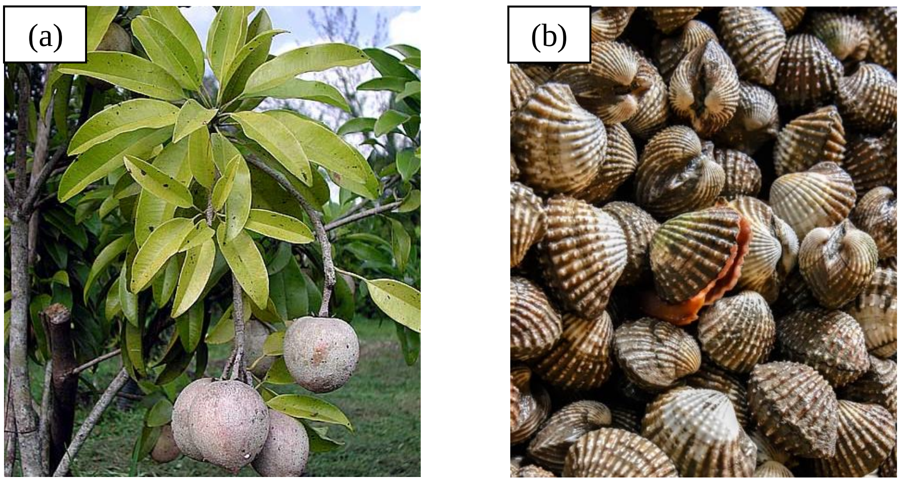

Figure 1.1 (a) Sapodilla (*Manilkara zapota L.*) leaves and (b) Cockles (*Anadara Granosa*)

1.2 Statement of Problem

In this era, heavy metal pollutants in water should be taken into account due to their toxic effects and potentials for substantial and long-term accumulation in sediments and organisms (Hua et al., 2012). Overwhelmed of heavy metals in the aqueous solution can be initiated by human activity in many areas such as industries (metallurgical, mining, chemical, tannery, battery, nuclear, iron, steel, leatherworking, photography, and electric appliance manufacturing), agriculture, shipping and others (Burakov et al., 2018). It is calculated that the annual global release of heavy metal reached 22,000 tons (metric tons) for cadmium, 939,000 tons for copper, 783,000 tons for lead and 1,350,000 tons for zinc (Abdolali et al., 2017). Heavy metal pollutants can cause disease if it accumulates in human body. The symptoms can be high blood pressure, speech disorders, fatigue, sleep disabilities, aggressive behaviour, poor concentration, irritability, mood swings, depression, increased allergic reactions, autoimmune diseases, vascular occlusion, and memory loss (Ihsanullah et al., 2016).

Moreover, lead (Pb) is one of the most common and the most toxic heavy metal pollutants. Pb can affect human by entering human body through the digestive tract and respiratory tract, and then goes into the blood circulation in the form of soluble salts, protein complexes or ions, etc (Ma et al., 2018). Pb is strongly pro-organizational. It affects and damages many of the body organs and systems, such as kidney, liver, reproductive system, nervous system, urinary system, immune system and the basic physiological processes of cells and gene expression (Su et al., 2014).

According to standard set by Malaysian Department of Environment, the portion of Pb that can be tolerated in water is around 0.5 mg L^{-1} (Hamdzah, 2018). In fact, the concentration of lead in wastewater is found at high level at 329 mg L^{-1} at Selangor River near by a mining site (Sakai et al., 2017). Similarly, Pb concentration at industrial area, Port Klang was found with highest quantity 128 mg L^{-1} (Yunus et al., 2020). Moreover, Pb concentration in water samples of several areas in Langkawi Island was at range 1.58 to 4.50 mg L^{-1} (Octavianti and Jaswir, 2017). In addition, investigation by Department of Environment Malaysia (DOE) in 2015 revealed that about 168 rivers were slightly polluted and 33 rivers were polluted out of 477 monitored rivers around Malaysia (Prabhakaran et al., 2017).

Additionally, Who Health Organization (WHO) has set the standard portion of Pb in surface water around 0.1 mg L^{-1}. The existence of heavy metal pollutants will also become a threat to the availability of clean water resources with around 40% of the population are facing the water crisis issues (Carolin et al., 2017). According to WHO data, 1.1 billion people worldwide still find difficulty to access to adequate drinking water sources, while about 2.4 billion people do not have access to basic sanitation (Vunain et al., 2016).

Likewise, WHO has also estimated that about 1.6 million of people die each year due to water-related diseases with 90% among them are children under age of 5 years old. In line with this, it is forecasted by World Water Council that by the year of 2030, about 3.9 billion people will live in region which is lack of water. Although, the existence of metals are necessary in trace quantities, but can be threats to environmental and human health at high concentrations (Xu et al., 2018). Additionally, based on United Nations (UN), 80% of all industrials wastewater in the developing countries is released to the environment without any prior treatment (Joseph et al., 2019)

Thus, the removal of lead from wastewater becomes major concern in order to overcome heavy metal contamination problems and to avoid further detrimental effects. The most common technique of heavy metal remediation is adsorption using commercial activated carbon. However, industrial users are reluctant to apply activated carbon for removing heavy metals from wastewater because of the expensive of chemical treatment as well as consume high energy for the activation process (Asuquo and Martin, 2016). Activated carbon also has lower removal efficiency due to it random pore geometry that causes difficulty for pollutants to reach it active sites (Da'na, 2017). Beside, activated carbon adsorbent also requires posttreatment due to the production of toxic sludge (Zhou et al., 2018). Moreover, activated carbon cannot be recycled and regenerated (Farooq et al., 2010). Therefore, this study will mainly focus on developing high capacity, and eco-friendly adsorbents derived from biomass waste that can be utilized in reducing the quantity of Pb(II) as contaminants in water.

1.3 Objective of Study

The main aim of this research is to develop green adsorbents that can adsorb high quantity of Pb(II) from aqueous solution. The objectives of this research are:

1. To synthesize Fe/C/Al_2O_3, Fe/C/M.S, and Fe/C/C.S adsorbents for adsorbing Pb(II) from aqueous solution.
2. To investigate the adsorbents activity by optimizing the concentration of Pb(II), dosage of adsorbent, and contact time.
3. To investigate the isotherm and kinetic study of adsorbents.
4. To characterize the best prepared adsorbents in order to understand the chemical and physical properties of the adsorbents.

1.4 Scopes of Study

This research emphasized on adsorbing heavy metal lead (Pb(II)) from aqueous solution using alumina beads (Al_2O_3), molecular sieve type 3Å-beads, 4–8 mesh (M.S), and cockle shell (C.S) based adsorbent which was coated by iron (Fe) and carbon (C) through wetness impregnation method and labelled as Fe/C/Al_2O_3, Fe/C/M/S, and Fe/C/C.S. Fe was obtained from iron salts ($Fe(NO_3)_3.9H_2O$), whereas C.S ($CaCO_3$) was retained from calcined crushed cockle shell. Meanwhile, C was derived from calcination dried sapodilla leaves extract. Then, adsorbents activity was investigated on the concentration of Pb(II) range 20–150 mg L^{-1}, dosage of adsorbent range from 0.5 to 2.5 g, and contact time for 4 h. Also, the reusability at optimum condition of potential adsorbent was done in four cycles while the stability was investigated through leaching test by X-ray fluorescence (XRF) as well as the effectiveness of C from sapodilla leaves extract as the binder agent.

Next, the potential adsorbents obtained was characterized using various techniques in order to understand the properties of the adsorbents such as X-ray diffraction (XRD) analysis for bulk structure, nitrogen adsorption-desorption (NA) for pore distribution and surface area of the adsorbents and field emission scanning electron microscope-energy dispersive X-ray (FESEM-EDX) for morphology and elemental composition study, fourier-transform infrared spectroscopy (FTIR) for functional group and other chemical elements analysis. Lastly, The interaction between Pb(II) pollutants and the adsorbents was carried out by isotherm study through Langmuir and Freundlich models, and the kinetic study through pseudo-first order, pseudo second order.

1.5 Significance of Study

Adsorption is one of the techniques that are more versatile and widely used for the removal of aqueous heavy metals compared to others. The use of adsorption also shows significant result in environment since it helps deescalating heavy metal contaminants in water. By developing the use of adsorbents for overcome heavy metal pollution issue in water will also affect the accessibility of clean water that becomes major concern in some areas all around the world in the past decades.

Among methods that have been applied to overcome heavy metal contamination problems, adsorption has proved to be a promising technique because the ease of operation and comparable low cost of application in treatment process. In addition, adsorption renders several merits such as simple utilization, economical, good pH tolerance, high efficiency and selectivity, and can be applied as large industrial processing capacity. Besides, the use of $CaCO_3$ as adsorbents derived from cockle shells will also benefit in taking down problems in mussel shells waste as well as use of sapodilla leaves improves has benefit in reducing the waste.

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