UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF DURIAN PEEL-BASED ADSORBENT FOR ZINC AND LEAD IONS REMOVAL

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By

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Thesis Submitted to the School of Graduate Studies, University Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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September 2020

Chairman : Associate Professor Datin. Ir. Siti Aslina Hussain, PhD
Faculty : Engineering

Toxic waste discharged into water bodies by anthropogenic activities is a worldwide problem. In relative to Malaysia, several reports of surface water contamination are from toxic waste discharged by manufacturing industries. For instance, the Juru and Langat rivers, Johor Straits, the marine environment of Port Klang, etc. were reportedly contaminated by toxic pollutants, including heavy metals. Similarly, discovery of several heavy metals on clam species from 34 different sites on the Malaysian coast revealed severe contamination. Ironically, Pb^{2+} and Zn^{2+} were categorically listed among the significant issues of concern in Malaysia. Therefore, the main objective of this research is to treat excess Pb^{2+} and Zn^{2+} concentrations from aqueous medium using biosorbent developed from durian waste. The basic concept is that "a waste" was applied to treat "a waste" sufficiently. Also, durian waste was transformed into a value-added biosorbent using physical, chemical, and thermal activations. The two prepared adsorbents were termed as hydrochloric acid-modified durian peel (HAMDP) and durian peel activated carbon (DPAC). Characterization of biosorbents such as particle size, SEM, BET surface area analysis, ATR-FTIR, elemental analysis, and pH_{PZC} was performed. Subsequently, the application of adsorption studies in removing Pb^{2+} and Zn^{2+} in static (batch adsorption) and dynamic (fixed-bed column) systems was achieved. The wastewater comprises mono-component (in a batch process) and bi-component or multi-metal ion (in fixed-bed column) systems. Real wastewater from the textile industry was also treatable using these biosorbents. The results showed that non-modified durian peel (NMDP), HAMDP, and DPAC have an average particle size of 206.7, 110.4, and 5.6 µm, respectively. BET surface area analysis revealed that NMDP, HAMDP, and DPAC have a surface area of 0.6793, 0.8807, and 9.1480 m^2/g. Morphologically, the improvement of the biosorbent porosity was testified by SEM. The ATR-FTIR analysis advised the involvement of several functional groups such as hydroxyl, aliphatic, carbonyl, aromatic amines, etc. as an active site on the biosorbents that influence the adsorption mechanisms. The elemental analysis also indicated the fruitfulness of durian peels as a potential precursor for activated carbon production. Batch biosorption study revealed that the influential parameters are pH (optimum: 8.0 for Zn and 7.0 for Pb), temperature (optimum: 40 °C for Zn and 50 °C for Pb), contact time (optimum: 240 min for Zn and 60 min for Pb) with maximum adsorption capacities of 36.7 and 127.83 mg/g for Zn and Pb, respectively. Biosorbent dose of 0.5 g in 100 ml is sufficient in this study. The isotherm studies were then fitted to Langmuir and Freundlich models. Desorption and regeneration studies showed the reusability of the biosorbents. Furthermore, column studies on single and multi-metal ion systems using DPAC indicated the capability of the biosorbent in treating excess Zn and Pb in the real industrial wastewater. The process fitted to Langmuir and linear driving force (LDF) methods. Finally, real wastewater from the textile industry was tested to validate the biosorbents. The worthwhile novelty is the easiest way of an activated carbon production process suggested in this work to achieve acceptable performance of durian peels. Hence, the biosorbent applied in this research can treat heavy metals such as Zn^{2+} and Pb^{2+} in Malaysia's real industrial effluent.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN BERDASARKAN-KULIT DURIAN UNTUK PENYINGKIRAN ADSORBEN ZINK DAN PLUMBUM IONS

Oleh

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Pembuangan sisa toksik ke dalam kumpulan air kerana aktiviti antropogenik menjadi masalah di seluruh dunia. Di Malaysia, terdapat beberapa laporan mengenai pencemaran permukaan air akibat pembuangan sisa toksik dari industri pembuatan. Contohnya, Sungai Juru dan Langat, Selat Johor, persekitaran laut Pelabuhan Klang dan lain-lain dilaporkan tercemar disebabkan oleh bahan pencemar beracun yang mengandungi logam berat. Begitu juga dengan beberapa kajian logam berat pada spesies kerang di 34 lokasi berbeza di pantai Malaysia yang menunjukkan pencemaran yang teruk. Ironinya, Pb^{2+} dan Zn^{2+} dikategorikan antara isu utama yang menjadi perhatian di Malaysia. Oleh itu, objektif utama penyelidikan ini adalah untuk merawat kepekatan lebihan Pb^{2+} dan Zn^{2+} dari medium berair menggunakan biosorben yang dihasilkan daripada sisa durian. Konsep dasarnya adalah sisa digunakan untuk merawat sisa secukupnya. Di samping itu, sisa durian diubah menjadi biosorben yang bernilai menggunakan fizikal, kimia dan pengaktifan haba. Kedua-dua adsorben yang telah disiapkan dan dibangunkan dikenali sebagai asid hidroklorik pengubah suai kulit durian (HAMDP) dan kulit durian karbon aktif (DPAC). Pencirian biosorben seperti saiz zarah, SEM, analisis luas permukaan BET, ATR-FTIR, analisis elemen dan pHPZC dilakukan. Selepas itu, diaplikasi didalam kajian penjerapan dalam penyingkiran Pb^{2+} dan Zn^{2+} dalam keadaan sistem statik (kumpulan penjerapan) dan sistem dinamik (kolum lapisan tetap) dicapai. Air sisa terdiri daripada sistem mono-komponen (dalam proses kumpulan) dan dwi-komponen atau multi-ion logam (kolum lapisan tetap). Air sisa daripada industri tekstil dirawat menggunakan biosorben yng terhasil ini. Hasil kajian menunjukkan bahawa kulit durian yang tidak diubah (NMDP), HAMDP dan DPAC mempunyai purata saiz zarah masing-masing 206.7, 110.4 dan 5.6 μm. Analisis luas permukaan BET menunjukkan bahawa NMDP, HAMDP dan DPAC memiliki luas permukaan 0.69393, 0.8807 dan 9.1480 m^2 / g. Secara morfologi, penambahbaikan keliangan biosorben dibuktikan oleh SEM. Melalui analisis ATR-FTIR terdapat penglibatan beberapa kumpulan berfungsi seperti hidroksil, alifatik, karbonil, amina aromatik, dan lain-lain sebagai tapak aktif

biosorben yang mempengaruhi mekanisme penjerapan. Analisis elemen juga menunjukkan buah kulit durian yang subur sebagai pendahulu yang berpotensi untuk pengeluaran karbon aktif. Kajian biosorpsi kumpulan menunjukkan bahawa parameter berpengaruh adalah pH (optimum: 8.0 untuk Zn dan 7.0 untuk Pb), suhu (optimum: 40 untuk Zn dan 50 untuk Pb), masa hubungan (optimum: 240 untuk Zn dan 60 untuk Pb) dengan maksimum kapasiti penjerapan masing-masing 36.7 dan 127.83 mg / g untuk Zn dan Pb. Dos biosorben 0.5 g / 100 ml adalah mencukupi dalam kajian ini. Kajian isoterma sesuai dengan model Langmuir dan Freundlich. Kajian desorpsi dan regenerasi menunjukkan kebolehgunaan semula biosorben. Selanjutnya, kajian kolum menggunakan sistem ion tunggal dan multi-ion logam menggunakan DPAC menunjukkan kemampuan biosorben dalam merawat lebihan Zn dan Pb dalam air sisa industri. Proses ini sesuai dengan kaedah Langmuir dan daya pendorong lelurus (LDF). Akhirnya, air sisa sebenar dari industri tekstil diuji untuk mengesahkan biosorben. Kebaharuan yang bermanfaat adalah kaedah termudah dalam proses pengeluaran karbon aktif yang dicadangkan dalam kerja ini untuk mencapai prestasi kulit durian yang dapat diterima. Oleh itu biosorben yang digunakan dalam penyelidikan ini mampu merawat logam berat seperti Zn^{2+} dan Pb^{2+} dalam sisa kumbahan perindustrian di Malaysia.

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LIST OF ABBREVIATIONS

DOE	Department of Environment
WHO	World Health Organization
MARDI	Malaysian Agricultural Research Development Institute
Kt	Kilotons
AAS	Atomic Absorption Spectrometer.
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
A.R	Analytical Reagent
ATR-FT-IR	Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy
HAMDP	Hydrochloric acid modified durian Peels
DPAC	Durian Peels Activated Carbon
RDP	Raw Durian Peel
NMDP	Non-modified durian peels
OVAT	One Factor At a Time
AWB	Agro-based Waste Biosorbent
HM	Heavy Metal
MCLs	Maximum Contamination Levels
USEPA	United States Environmental Protection Agency
COD	Chemical Oxygen Demand
BOD	Biological Oxygen Demand
D.I	Deionized
IUPAC	International Union of Pure and Applied Chemistry
SEM	Scanning Electron Microscopy
SAM	Solid Additional Method
BET	Brunauer Emmett Teller
SSE	Sum of Squares Error

LIST OF NOTATIONS AND GLOSSARY OF TERMS

α	initial sorption rate	mg/g-min
b	energy constant related to the heat of biosorption	L/mg
B_T	Temkin constant related to the biosorption energy	kJ/mol
β	surface coverage and activation energy for chemisorption	g/mg
C_e	adsorbate concentration at equilibrium	mg/L
C_o	initial adsorbate concentration	mg/L
C_{Ae}	equilibrium concentrations of adsorbate on biosorbent	mg/L
D_i	internal diameter	mm
h	Initial sorption rate.	mg/g-min
k_1, k_2	pseudo-first and second-order rate constant	1/min
K_T	binding constant at equilibrium	L/g
K_f	Freundlich constant related to bonding energy	$(mg/g)(L/mg)^{(1/n)}$
nq_m	Freundlich exponent (surface heterogeneity) or number of observations	L/mg
q_e	monolayer capacity of the Langmuir isotherm	mg/g
$q_{e,cal}$	adsorbent phase concentration after equilibrium	mg/g
q_t	calculated biosorption capacities at equilibrium	mg/g
Q	biosorption capacity at time flow rate	mg/g
R_L	separation factor	mL/minute
R	universal gas constant	unit less
T	absolute temperature	8.314 J/mol-K
t	time	K
V	volume of solution	seconds/minute/hour
W	mass of biosorbent	L

y	yields/response	g
P_{zpc}	point of zero charge	(mg/g)
pH_i	initial pH	-
pH_f	Final pH	-
ΔpH	Difference in pH	-
ΔG^o	Gibbs free energy	-
ΔH^o	enthalpy change	kJK^{-1} mol^{-1}
ΔS^o	entropy change	J/KG
K*c*	Equilibrium constant	J/K
E_a	activation energy	KJ mol^{-1}

CHAPTER 1

INTRODUCTION

1.1 Background

Water pollution due to natural and anthropogenic activities is inevitably an issue of global concern. Agricultural practices, household discharges, and industrial activities are primarily responsible for aquatic pollution. Generally, pollutants generated from industries are classified into organic and inorganic contaminants (Carolin et al., 2017). Heavy metals (HMs) were characterized as one of the inorganic pollutants found from industrial effluents in a substantial amount. The definition of HMs being widely accepted by the scientific world is; the metal ions with a specific gravity that is at least five times higher than the specific gravity of water. Therefore, HMs have a particular weight of at least higher than 8 gm/cm^3 (Singh et al., 2018). The toxicity of metal ions was intensively discussed in the literature. The most common hazardous HMs include As, Cd, Cr, Hg, Pb, Ni, Zn, and Cu. They have deleterious impacts on flora and fauna, human health, and the environment due to possession of two primary characteristics, namely non-biodegradability and persistence (Zhang et al., 2017; Milojkovic et al., 2016; George et al., 2016). For instance, Malaysia has over 3200 manufacturing industries (https://www.mida.gov.my/; DOE, 2017), depending on its purpose, either for electroplating, semi-conductor, battery production, fertilizer or textile industries, can generate a substantial amount of heavy metals. Recently, numerous investigations revealed that Malaysian surface water had been contaminated by anthropogenic activities (see section 1.5). In Malacca and Johor Straits, several metal ions significantly affected the clam species. Both Pb^{2+} and Zn^{2+} were categorically stated as an issue of concern in Malaysian surface water, specifically rivers and marine environments (Sany et al., 2013a; Sany et al., 2013b; Sabri et al., 2014; Maadin et al., 2016). These Pb^{2+} and Zn^{2+} were investigated to be pollutants present in sediments of the Juru and Langat rivers.

Similarly, the concentrations of Zn^{2+} and Pb^{2+} in Johor Straits are 2 and 3 times higher than global shale values. Additionally, the marine environment of Port Klang is also significantly contaminated by Pb in sediment and Pb and Zn in water along with other metal ions (Sany et al., 2013a; Sany et al., 2013b). Discharged industrial effluent and port activities are primarily responsible for the contamination (Shazili et al., 2006; Sabri et al., 2014; Maadin et al., 2016).

The conventional methods currently applied in minimizing heavy metals from wastewater are precipitation, coagulation, membrane filtrations, reverse osmosis, electrochemical operation, etc. However, these techniques have some potential drawbacks such as high cost, inadequate treatment, sludge generation, inability to treat diluted heavy metals in solution, etc. Hence, the decontamination of excess metal ions through economical and effective sorbent is required. The adsorption technique was a good process that fulfilled such criteria (Chakraborty et al., 2020). Besides, adsorbents should be selected based on lower cost, availability in abundance, easily modified, adequate adsorption capacity, and has regeneration ability (Nguyen et al., 2013; Chakraborty et al., 2020; Alalwan et al., 2020; Igalo et al., 2020). Hence, durian peels were carefully selected as the precursor adsorbent in this work while keeping in mind its potentiality.

Moreover, Malaysia produces 376 kt of durian fruit every year (Aziz & Jalil, 2019). The edible portion of the fruit accounts for 15-30 % of the whole fruit. On average, 290 kilotonnes (kt) of durian waste is generated from the durian fruit industry, let alone other fruits. Indeed, waste generation is in bulk, and to avoid nuisance to the society and the environment at large, durian peels have to be appropriately utilized.

Since the durian peels are cellulosic material, the adsorbent is rich in active functional groups such as hydroxyl, aliphatic, carbonyl and amides (Kumar et al., 2020) that can attract Pb^{2+} and Zn^{2+} with a strong binding force to its surface. To the best of our knowledge, there was no report of durian peels being used as an adsorbent in treating heavy metals in real wastewater treatment. As far as a green technology concept is concerned, this research proposes the development of biosorbent from a waste (durian peel) in the treatment of wastes/pollutants (Pb^{2+} and Zn^{2+}). In the literature, durian peels were applied to treat various organic pollutants and a handful of HMs (see section 2.4.2). Though there are reports of durian peel wastes undergo some modification for performance improvements, the applied processes were quite tedious especially in the activated carbon production aspect. Simple HCl treatment and easily activated carbon production from durian peel with acceptable performance in Pb^{2+} and Zn^{2+} were observed in the current work.

These HMs are reportedly among the current significant issues going on in Malaysia. Therefore the purpose of selecting Zn^{2+} and Pb^{2+} as HMs in consideration were based on the literature survey (Sany et al., 2013a; Wan Maznah et al., 2012; Ab Razak et al., 2015; Beh et al., 2012; Shuhaimi-Othman et al., 2012; Hossen et al., 2015; Najiah et al., 2014; Abdullah et al., 2014; Shazili et al., 2006; Bashir et al., 2013). According to the Malaysian Department of Environment (DOE, 2010), the acceptable conditions for industrial or mixed Zn^{2+} effluent discharge is 2 mg/L for both standard A and B (Zwain et al., 2014). In comparison, Pb^{2+} is 0.1 and 0.5 mg/L for standard A and B, respectively. Similarly, the World Health Organization (WHO) stated 3 and 0.1 mg/L as the permissible limit for Zn^{2+} and Pb^{2+}, respectively, in drinking water (WHO, 2011). The primary sources of these HMs contaminations are industrial activities such as ceramics, fabrics, mining, metal coating, paints, deodorants, lead-acid battery, oils, fertilizer, metal phosphate, electronic and many more (Shanmugaprakash et al., 2018; Bhatnagar et al., 2015; Mishra , 2014; O'Connell et al., 2008; Sadeek et al., 2015; Srivastava et al., 2015; Vijayaraghavan & Balasubramanian, 2015; Smith et al., 2015; Iyer et al., 2015; Mittal et al., 2016, Ahmed et al., 2018, Wang et al., 2018). Beyond the acceptable threshold, these heavy metals cause numerous diseases, especially to the central and peripheral nervous systems (Malamis & Katsou, 2013; Zhang et al., 2017; Wang et al., 2018; Iyer et al., 2015). Thus, given such harmful effects, the minimization of HMs, specifically Zn^{2+} and Pb^{2+}, as far as this study is concerned, is essential to adequately safeguard our life and the environment at large.

Numerous wastewater treatment techniques (see Section 2.5: Table 2.2) have been employed by several researchers to detoxify contaminated aqueous medium, mostly from industrial effluents, to protect our water sources. Conventional methods adopted for removal of heavy metals include precipitation, coagulation or flocculation, ion exchange, membrane filtration, reverse osmosis, complexation/sequestration, electrochemical operation, and biological treatments (Vijayaraghavan & Balasubramanian, 2015; Mittal et al., 2016; Zhang et al., 2017; Nyairo et al., 2018). These methods have inevitable drawbacks (see Section 2.5; Table 2.2); for instance, if the content of HMs is very minimal in large water bodies, such methods will not be suitable, mainly due to cost. Adsorption technique happen to be one of the biological treatments reported by prominent researchers in this field as a capable process to treat HMs from wastewater (Foo & Hameed, 2011; Volesky, 2003; Volesky & Holant, 1995). Agricultural and industrial wastes as adsorbents have been rigorously studied as fruitful non-conventional approaches in treating metal ions from contaminated solutions. In the literature, available materials reported in biosorption of Zn^{2+} and Pb^{2+} include *Caryota urens* seeds (Ravulapalli & Ravindhranath, 2018), activated carbon derived from *styrax officinalis* seeds (Sellaoui et al., 2016), several activated carbons (Largitte & Pasquier, 2016), sugarcane bagasse (Krishnan et al., 2016), olive mill solid waste (Abdelhadi et al., 2017), banana peels and cauliflower leaves (Ahmad et al., 2018) orange peels (Feng et al., 2011), Tamarind wood (Acharya et al., 2009). A biosorption phenomenon is a process whereby pollutants are adsorbed onto biomass (biological materials). It is generally applied to the vast body of wastewater containing a low toxic metal ions concentration (Barakat, 2011; Gunatilake, 2015; Akpor et al., 2014; Ayangbenro & Babalola, 2017). This technique's significant advantage is the use of inexpensive and renewable materials that are abundantly available as waste (usually from agro-based industry) to reduce or possibly eliminate HMs concentration in contaminated water.

1.2 Biosorbents from Agricultural Waste/Biomass

The term "biomass" refers to materials obtained from living organisms, i.e. plants and animals. For instance, agricultural wastes, wood, short-rotation woody crops, bagasse, waste paper, sawdust, bio-solids, grass, and food processing waste, aquatic plants, animal waste, red algae (*Ceramium virgatum*), neem (*Azadirachta indica*), food waste (areca), orange peel, etc. (Demirbas, 2008). Durian waste is also a typical biomass example, which is abundantly available as waste material in Malaysia. It is the biosorbent applied throughout this study to treat Zn^{2+} and Pb^{2+} in synthetic and raw industrial wastewater (from the textile industry).

1.3 Problem Statement

Industries such as battery production, paints, fertilizer, and mining activities, discharge their effluents containing substantial amounts of HMs into the environment. Such metals include cadmium, lead, chromium, mercury, zinc, nickel, copper, and iron (Salam et al., 2019; Singh et al., 2018; Zhang et al., 2017; Milojković et al., 2016; George et al., 2016), which are channelled into the marine environment as contaminants by anthropogenic activities. The release of such wastewater into water bodies could result in deteriorating drinking and groundwater quality. The HMs are non-biodegradable, persistent, and toxic that show a detrimental effect on the eco-system. Examples of HMs incidents are well documented in the literature. In Japan, Minamata and Itai-Itai outbreaks are typical examples of HMs toxicity (Chen et al., 2015; Kaji, 2012). Consumption of seafood such as fish from a water source contaminated by mercury was the prime root cause for Minamata disease. Whereas Itai-Itai disease was due to cadmium poisoning, and the issue drew global attention. These HMs such as Pb^{2+} and Zn^{2+} [as the primary concerning HMs in the present work] could cause muscular stiffness, pancreas damages, diarrhoea, lethargy, headache, hallucinations, anemia, and ischemic heart disease when consumed above acceptable limit, especially in drinking water. Even though Zn^{2+} and other HMs such as Cu^{2+} and Fe^{3+} are essential elements for human growth, they instigate side effects if found in excess exposure. In Malaysia, Pb^{2+} and Zn^{2+} were investigated as contaminants found in Juru and Langat sediments (Sany et al., 2013a; Shazili et al., 2006).

Similarly, the concentrations of Zn^{2+} and Pb^{2+} in Johor Straits are 2 and 3 times higher than global shale values. Additionally, the marine environment of Port Klang is also significantly contaminated by Pb^{2+} in sediment and Zn^{2+} in water (Sany et al., 2013a; Sany et al., 2013b) along with other contaminants. Discharged industrial effluent and port activities are the main root causes for the contamination. Furthermore, the study on six HMs namely: Cd, Pb, Ni, Cu, Zn, and Fe in different clam species from 34 sites on the Malaysian coast revealed HM contaminations (Hossen et al., 2015). Ironically, in Malaysia, Pb^{2+} and Zn^{2+} were mentioned as significant concerns (Sabri et al., 2014; Maadin et al., 2016). That prompted us to select them as HMs of consideration in this work. Therefore, considering such issues in Malaysia, it is crucial to treat these HMs and reduce the contamination level to a permissible limit, right from the source, especially from industrial effluents prior to disposal into water bodies (Zhang et al., 2017). This research aims to reduce, if not to eliminate, the amounts of Zn^{2+} and Pb^{2+} present in industrial wastewater down to an allowable limit recommended by prominent standards prior to disposal. These two HMs were treated using a process known as the "Biosorption Technique". Biosorbent adopted in this work is "durian peels." The average weight of durian fruit is approximately 1.5 kg (3.3 lb). The edible portion of the fruit accounts for merely about 15-30 % of the whole fruit. Since Malaysia produces 376 kt of durian fruit every year, that implied 263.2 to 319.6 kt of organic waste are generated as a byproduct from the durian fruit industry alone annually. The resultant large quantity of durian waste accounts for the feasibility of selecting it as biosorbent in this work. Durian peels are rich in cellulose (60.45 %), lignin (15.45 %) and hemicellulose (13.09 %) (Cheok et al., 2018), which resulted in abundant availability of active sites such as OH, NH, CO functional groups, among others. These groups have a good capability to attract HMs on the surface of the biosorbent. Besides, proximate and ultimate analysis on durian peel reported its significant properties that are suitable for activated carbon production. Those properties are high carbon content, low ash content, negligible sulphur content, etc. (Nazem et al., 2020; Ozsin et al., 2019). Moreover, majority of reported articles used durian peels as adsorbent in treating other pollutants such as dye, nitrates and phosphates. Exploration of HMs using durial peels as precursor adsorbent is lacking in the literature. Therefore, this research is performed to fill the missing knowledge gap.

Durian waste (peel/shell/husk/rind) was collected from local traders along the streets of Serdang in Selangor and prepared for the adsorption process. Untreated fruit peels and vegetable waste have implications of lower adsorption capacity of pollutants and could increase unwanted substances into the water bodies during application (Patel, 2012; Nguyen et al., 2013). Before applying wastewater treatment, it was highly recommended to pre-treat or modify agricultural/fruit wastes such as durian peels (Schwantes et al., 2018). Consequently, HAMDP and DPAC were carefully prepared and adopted as adsorbents in this work. Synthetic wastewater of single metal system [containing either Zn^{2+} or Pb^{2+}] and multi-metal ion system [with the constituent of Zn^{2+}/Pb^{2+}] along with other elements, are considered in this work.

Similarly, real industrial textile wastewater was tested for validation purposes using the biosorbents. Such two adsorbents were not recorded enough for Pb^{2+} and Zn^{2+}. This work's outcome is aimed in developing cost-effective methods to prepare and apply eco-friendly biosorbents with a rational hypothesis.

1.4 Hypothesis

Generally, this study is designed to assess the hypothesis that durian peels can perform effectively in treating Pb^{2+} and Zn^{2+} in wastewater. Moreover, the biosorbent produced from durian peel is easy to use, ecologically friendly, has sufficient adsorption capacity, and is a low-cost material. Subsequently, the following additional assumptions were considered:

- Chemically modified durian peel (using acid or alkaline) can enhance the sorption performance of Pb^{2+} and Zn^{2+} from contaminated water bodies compared to non-modified durian peels.
- Based on the literature, acid modified durian peel could perform better than alkaline modified durian peels to treat heavy metals from an aqueous medium.
- Durian peel can be a good precursor for activated carbon production due to its high percentage in cellulose composite, high carbon content, and low ash content as its components.

- The most influential factors affecting batch studies' adsorption process can be solution's pH, initial metal ion concentration, contact time, biosorbent dose, reaction temperature, agitation speed, and nature of biosorbent.
- The most influential parameters affecting fixed-bed column studies were bed height, initial metal ion concentration, and flow rate.
- The mechanism involved in the adsorption of Pb^{2+} and Zn^{2+} can be a combination of ion exchange, complexation, and adsorption complexation.
- The biosorbents can be regenerated and reused several times.

1.5 Research Objectives

1) To investigate the potentials of HAMDP and DPAC on biosorption of Zn^{2+} and Pb^{2+} ions using batch studies.
2) To evaluate the capability of DPAC on sorption of Zn^{2+} and Pb^{2+} ions in bicomponent/multi-metal system using dynamic (fixed-bed column) process.
3) To validate the applicability of HAMDP and DPAC in treating real industrial effluent from the textile industry.

1.6 Research Scope

This research's main focus was to develop a cost-effective adsorbent to treat heavy metal ions from wastewater. Durian peel (precursor adsorbent) that had physical, chemical, and thermal treatments will transform its natural state (waste) into value-added material (fruitful adsorbent). The biosorbents were characterized for active surface sites, surface area analysis, particle size analysis, surface morphology, proximate and ultimate analysis to identify the mechanisms involved in the adsorption process. The sorption process is only limited to single (synthetic), binary (synthetic), and multi-metal (real and simulated) ion system of Zn^{2+} and Pb^{2+} from wastewater using batch (static) and fixed-bed column (dynamic) studies. The single metal-ion method was performed separately for Zn^{2+} and Pb^{2+} to identify the adsorption possibility of the adsorbents without the hindrance of other pollutants. It is the common practice applied in the literature. For the batch system, several parameters were considered, including the effect of pH (range from 2-12); effect of contact time (ranges between 1 minute to 48 hours in both cases), the effect of initial metal ion concentrations (ranges between 10 to 250 mg/L in both cases), the effect of temperature (30 to 50 °C) and the impact of biosorbent dose (0.5 – 2 g) at agitation speed (120 rpm). In the dynamic system, the experimental conditions considered are bed height (DPAC only) of 10 cm, a volumetric flow rate of 1 mL/min, the weight of biosorbent is c.a 4.25 g, and initial metal ions concentration of 10-250 mg/L. Numerous modeling techniques including isotherm, kinetic, thermodynamic and breakthrough [Langmuir type, and linear driving force (LDF) models], were incorporated. The selection of appropriate material was made for the studies based on the literature survey. In column studies, the experimental conditions were implemented based on the literature review as well. The adsorption process was applied to remove excess Zn^{2+} and Pb^{2+} from contaminated water, i.e. synthetic and real industrial wastewater. Hence, this study does not include the treatment of other pollutants than Zn^{2+} and Pb^{2+}. For validation purposes, the researcher had drawn a boundary for this work to include the treatment of textile industrial effluents collected from a particular Malaysian industry. Hence, the idea of this research can be applied in all existing industrial effluents in Malaysia.

1.7 Contributions

The main contribution of this research is to investigate the potential of durian peels in treating heavy metals (Zn^{2+} and Pb^{2+} as far as this study is concerned) from an aqueous medium applicable to industrial wastewater decontamination. The critical point of observation is that materials that are entirely being wasted should be considered as the adsorbent's primary source. For instance, when the applied seeds (say durian fruit seed) for an activated carbon production were performed efficiently, researchers decided to scale up the application; indeed, scarcity of such seeds would be encountered. In anticipation of such challenges, exclusive wastes are highly recommended. The high carbon content, low ash content, and availability of cellulose composition of common durian waste biomass prompted it to be a fruitful precursor for activated carbon production with a reasonable grade. To the best of our knowledge, activated carbon prepared from durian peel has never been reported in real industrial effluent treatment. Also, very few articles are available on the treatment of HMs using durian peels as a precursor adsorbent.

Moreover, most reported articles use durian peels as adsorbent treating other pollutants such as dye, nitrates, and phosphates. Exploration of HMs using durian peels as precursor adsorbent is still lacking in the literature. Therefore, this research was aimed to fill the missing gap in knowledge. Desorption and regeneration studies were also performed to assess the reusability of the biosorbent. Thus, this research could significantly contribute in establishing the economically viable industrial column to treat HMs from Malaysian industrial effluents before disposal into water bodies. The worthwhile novelty is the easiest way for an activated carbon production process suggested in this work to achieve acceptable performance of durian peels.

1.8 Thesis Organization

This thesis is divided into five chapters, followed by appendices at the end. Chapter 1 describes a brief background on water pollution status and its effects on human beings, flora and fauna, and the environment. It also comprised the problem statements that provided some basis and rationale in identifying the present research direction to be followed. The objectives of the study were clearly stated, along with the scope to be considered. Chapter 2 covers the overview of adsorption's general concepts, utilization of biomass in adsorption of heavy metals. The review applies to the mechanism of biosorption phenomena, biomass/biological material, various agro-based biosorbents, factors affecting adsorption processes, the purpose of "modification and activation" of biomass, and finally, a summary of works in literature concerning the effect of parameters such as pH, temperature, initial metal concentration, etc. in biosorption of

Zn^{2+} and Pb^{2+} onto various agro-based waste biosorbents. The modellings of the biosorption process applied were also well described. Chapter 3 describes the materials and methods employed as well as biosorption characterization, modification, and activation methods adopted. Moreover, the conducted experimental procedures and sample analysis were well explained. Chapter 4 covers the overall results and discussion on the data obtained along with current relevant citations. Chapter 5 established the conclusions and future work recommendations from the findings of the present work.

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