POTENTIAL USE OF BIOCHAR FROM JACKFRUIT WASTE (*Artocarpus heterophyllus* lam) AS HEAVY METAL REMOVAL AGENT

MOHAMMED KADHEM ABID

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Thank you.

ABSTRACT

Conversion of agricultural waste products into biochar is regarded as one of several recycling and disposal options. The study aimed to use biochar from jackfruit waste as an agent for heavy metals removal. Jackfruit waste was selected in this study due to its abundance in Malaysia. The jackfruit waste was thermally activated by phosphoric acid at 500 ^{o}C. Analytical techniques including X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), surface area measurement (BET method), Raman spectroscopy, energy dispersive X-ray (EDX), and fourier transform infrared (FTIR) spectroscopy were used to characterize the synthesized biochars. The physicochemical characteristics of the synthesized biochars were examined using different conditions to determine their usefulness for heavy metals adsorption. The surface morphology of biochars revealed the distributions of numerous cavities, cracks and small pits over the surface with pore size ranged from 3.25 – 3.78 nm. The biochars were mainly macroporous with BET surface area of 3.64 m^2/g (biochar peel) and 4.25 m^2/g (biochar seed). The produced biochars displayed low cation exchange capacity (CEC) of 5.64±0.47 meq /g and 6.36± 0.05 meq /g with a bulk density of 0.7± 0.1 kg/cm^3 and 0.6±0.1 kg/cm^3. The best conditions for the removal of heavy metals were 0.1g biochar loading, 100 mg/L metal ions concentration and at pH 7. The adsorption process was spontaneously endothermal for the peel while exothermal for the seed. The experimental data fit well with the Langmuir isotherm model and the pseudo-second order kinetics. The maximum adsorption capacity of metal ions was in the sequence of Fe =4.40 mg/g < Pb=10.1 mg/g < Cu= 17.5 mg/g < Cd =20.0 mg/g < Mn =76.9 mg/g for peel and Fe =1.80 mg/g < Cd =52.6 mg/g < Mn =69.9 mg/g < Pb=76.9 mg/g < Cu= 116.7 mg/g for seed at pH 7. In conclusion, biochars synthesized from jackfruit wastes show the Langmuir isotherm and the pseudo-second order are the best-fitting models for heavy metals removal especially Cu (II) ions in wastewater. In implication, biochar from jackfruit waste may serve as an environmentally friendly heavy metals removal for the wastewater industry.

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ABSTRAK

Penukaran produk sisa pertanian kepada biochar dianggap sebagai salah satu daripada beberapa pilihan dalam proses kitar semula dan pelupusan. Kajian ini bertujuan untuk menggunakan biochar dari sisa nangka sebagai agen penyingkiran logam berat. Sisa nangka dipilih dalam kajian ini kerana ianya banyak terdapat di Malaysia. Sisa buah nangka telah diaktifkan secara termal menggunakan asid fosforik pada suhu 500°C. Teknik analisis termasuk pembelauan X-ray (XRD), mikroskopi pengimbasan pelepasan medan (FESEM), pengukuran kawasan permukaan (BET), Raman spektroskopi , tenaga dispersive X-ray (EDX) dan spektroskopi inframerah transformasi Fourier (FTIR) digunakan untuk mencirikan biochar yang disintesis. Ciri-ciri fizikal kimia biochar yang disintesis telah diuji menggunakan keadaan yang berbeza untuk menentukan kegunaannya bagi penjerapan logam berat. Permukaan morfologi biochar mendedahkan kepelbagaian kaviti, keretakan dan lubang kecil di permukaan dengan saiz liang antara 3.25 - 3.78 nm. Kebanyakkan biochar adalah makropori dengan luas permukaan BET iaitu 3.64 m^{S}/g (kulit biochar) dan 4.25 m^{2}/g (biji biochar). Biochar yang dihasilkan menunjukkan kapasiti pertukaran kation (CEC) rendah 5.64 ± 0.47 meq /g dan 6.36 ± 0.05 meq /g dengan ketumpatan pukal 0.7 ± 0.1 kg/cm^{3} dan 0.6 ± 0.1 kg/cm^{3}. Keadaan terbaik untuk penyingkiran logam berat adalah pada muatan biochar 0.1g, kepekatan ion logam 100 mg/L dan pada larutan pH 7. Proses penjerapan secara spontan adalah bersifat endotermal untuk kulit manakala eksotermal untuk biji. Data eksperimen bersesuaian dengan model isoterm Langmuir dan kinetik urutan pseudo-kedua. Kapasiti penjerapan maksimum ion logam adalah dalam urutan Fe = 4.40 mg/g <Pb = 10.1 mg/g <Cu = 17.5 mg/g <Cd = 20.0 mg/g <Mn = 76.9 mg/g untuk kulit dan Fe = 1.80 mg/g <Cd = 52.6 mg/g <Mn = 69.9 mg/g <Pb = 76.9 mg/g <Cu = 116.7 mg/g untuk biji pada pH 7. Kesimpulannya, biochar yang disintesis dari sisa nangka menunjukkan isoterm Langmuir dan urutan pseudo-kedua adalah model yang paling sesuai untuk penyingkiran logam berat terutama ion Cu (II) dalam air sisa. Secara implikasinya, biochar dari sisa nangka boleh berfungsi sebagai penyingkiran logam berat yang mesra alam untuk industri air sisa.

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AAS	Atomic adsorption spectrometer
AC	Activated carbon
Al	Aluminium
BET	Brunauer-Emmett-Teller
C (%)	Carbon ratio
Ca	Calcium
Cd	Cadmium
CEC	Cation Exchange Capacity
C_e	equilibrium concentration
Cu	Copper
DI	Deionized water
EC	Electrical conductivity
EDX	energy dispersive X-ray
Fe	Iron
FESEM	Emission Scanning Electron Microscope
FTIR	Fourier transform infrared spectroscopy
H (%)	Hydrogen ratio
H/C	Ratios hydrogen/carbon
IBI	International Biochar Initiative

K	Potassium
Mg	Magnesium
Mn	Manganese
N (%)	Nitrogen ratio
N/C	Ratio nitrogen/carbon
O	Oxygen
O/C	Ratio oxygen /carbon
Q_{max}	Maximum adsorption capacity
P	Phosphorus
Pb	lead
PH	Hydrogen Ion
S	Sulphur
S (%)	Sulphur ratio
SD	Standard Deviation
Si	Silicon
TGA	Thermogravimetric analysis
WHO	world health organization
XRD	X-ray diffraction
ΔS^o	Entropy
ΔG^o	Free energy change
ΔH^o	Enthalpy change

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Environmental pollution is considered as one of the major concerns worldwide. Pollution is mainly caused by various human activities related to rapid development in urban and industrial sectors where different pollutants contaminate the immediate environment. These include air pollution (emission of hazardous or toxic gases detrimental to human health), land pollution (usually results from the dumping of metal and plastic scraps which take several years for degradation) and water pollution (commonly caused by several industrial activities connected to discharge of chemicals). Water is contaminated by industrial effluents and other agricultural practices. In this regard, micropollutants such as heavy metal ions are major environmental concerns that affect terrestrial and aquatic biota. Both natural and anthropogenic sources contribute significantly to the elevated levels of metal in water. However, more recent research findings have linked water pollution to human activities due to the rise in industrialization (Zheng & Shi, 2017). Heavy metals are the most abundant form of pollution in Malaysia either in the form of solid or liquid. With vast industrialization and economic development in coastal regions, heavy metals continue to be introduced to the estuarine and coastal environments which eventually end up in rivers, runoffs, and land-based areas (Yu et al., 2008). Despite the environmental controls being implemented by Malaysian governmental agencies, contaminants from smaller, sometimes illegal industries are still extensive and represent the largest environmental problem (Tariq et al. 1996).

Pollutants that originate from various human activities are very dangerous for living creatures and thus, require immediate prevention through different technologies. Moreover, various heavy metals as pollutants that are leached out from the industries and mines can cause freshwater scarcity. Consequently, the growing population worldwide is subjected to a threat of clean water shortage for the survival in the near future. To overcome this problem, sustainable techniques have been demanded for the treatment of heavy metal pollutants to save and clean the environment and ground water. Several techniques have been introduced to treat such hazardous heavy metals pollutants present in ground water. However, most of the water treatment techniques need extensive knowledge and operational skills (Ding et al., 2016).

Conventional methods such as ion exchange, membrane filtration, chemical precipitation, solvent extraction, and electrochemical treatment (Mohammadnezhad et al., 2017) are available for eliminating heavy metals from industrial wastewater. These techniques have inherent limitations and collateral effects, such as high cost of operations, secondary pollution due to the addition of chemicals in the treatment, and low efficiency (Ding et al., 2016). Adsorption method is regarded as the most sustainable for the treatment of water contaminated with heavy metals and other pollutants. Owing to its ease of operation and the availability of diverse adsorbents, biomass-based adsorption method has become popular. It can also be used to remove various pollutants from water such as soluble, inorganic, insoluble organic, and even biological contaminants. Despite these benefits, usage of adsorption method is limited in industrial scale applications.

This shortcoming is attributed to the deficiency of suitable adsorbents with excellent adsorption capacity and absence of columns with commercial scale. For each pollutant, there must be a specific adsorbent to enhance the pollution treatment efficiency. Upon comparing the existing water treatment technologies with adsorption in terms of cost effectiveness, the following results were obtained; Adsorption > Evaporation > Aerobic > Anaerobic > Ion exchange > Electro-dialysis > Micro- and Ultra-filtration > Reverse osmosis > Precipitation > Distillation > Oxidation > Solvent extraction (Imran, 2012). The outcome showed that despite the limitations associated with adsorption technique, it is still the most viable treatment method for waste water. Biochar, a solid material obtained from thermochemical conversion of biomass in an oxygen-limited environment, has attracted widespread attention in environmental management and agricultural practices due to its potential benefits in carbon sequestration, contaminated water treatment, and soil amendments (Lian & Xing, 2017; Saadat et al., 2018).

Various agricultural waste materials such as oil palm, durian, and rambutan waste have been used to synthesize these adsorbents. Being abundant and free of cost, these materials are considered as sustainable adsorbents for the effective removal of pollutants from waste water. Motivated by these facts, present research used waste jackfruit seeds and peel to synthesize biochar adsorbent for the removal of heavy metals from contaminated river water in order to propose a sustainable adsorption technique. In this approach, one of the popular Malaysian agricultural waste biomasses, jackfruit peel and seeds, activated with biochar was synthesized as an adsorbent. This jackfruit-based adsorbent was utilized to determine its efficacy on heavy metal removal in different concentrations from water under variable contact times. The prepared adsorbent was characterized using different analytical techniques to evaluate its physiochemical properties. Influences of other adsorption parameters such as temperature, pH, contact time, and heavy metal concentration on the efficiency of the extracted biochar were tested for optimization.

1.2 Problem Statement

Industrial revolution and rapid urbanization due to growing population worldwide are regarded as the major cause for environmental pollution (Yadanaparthi et al., 2009). Water pollution is one of the most risky effluents that can cause several deadly diseases and illnesses (Ahmad et al., 2014). Research have revealed that heavy metals and other toxic chemicals from mining sites, mechanized farming sites, and other industrial activities are the main contaminants of water bodies worldwide (Ali & Aboul-Enein, 2005). Heavy metals when ingested in large concentration through the food chain can cause severe health issues such as cancer, cerebral problems, and renal failure to human beings (Smiciklas et al., 2006, Goswami & Purkait, 2014). Water pollution with potentially toxic elements (PTEs) has significantly increased over the last decades. PTEs including As, Cd, Cr, Cu, Hg, Ni, Pb, Se, vanadium (V), and Zn are responsible for health problems to the living creatures (Shaheen et al., 2018). Both natural processes and anthropogenic activities have led to elevated levels of PTEs on the surface and in the groundwater, waste streams, and effluents (Shaheen et al., 2018).

The United Nations Environment Programme and the United Nations World Water Assessment Programme reported that above 80% of sewage effluents and about 70% of industrial waste in some developing countries might be discharged without treatment into surface water, thereby polluting the usable water supply (Shaheen et al., 2018). Presently, heavy metal contamination arising from the naturally occurring abundant elements (found in the earth's crust and river bodies) appears as a serious threat (Ayangbenro & Babalola, 2017). In this rapid industrialization era, most of the health problems are associated with exposure of heavy metals that contaminate water consumption (Koki et al., 2018).

These heavy metals are responsible for the bioaccumulation and biomagnification in the food chain, causing even greater damage to the human health. Being the most important commodity, any scarcity of clean water in the near future may result in the incessant discharge of toxic industrial effluents into the water bodies (Haileslassie & Gebremedhin, 2015). Techniques such as electro-dialysis, reverse osmosis ion exchange, and freeze desalinization have been developed for treating heavy metals and other toxic materials that contaminate water (Imran Ali, 2012). Despite these techniques, an efficient and sustainable method for metal-contaminated wastewater treatment has yet to be developed. Existing techniques for the treatment of wastewater including chemical precipitation, membrane filtration, ion exchange, and electrochemical treatment suffer from various drawbacks related to secondary pollution, complexity of operations, and high operating cost (Fu & Wang, 2011; Zhang et al., 2017). Compared to these technologies, adsorption with the advantages of high efficiency, low cost, and simple operation process is generally considered to be one of the best options (Wan et al., 2016; Zare-Dorabei et al., 2016; Zhao et al., 2016). For the removal of heavy metals from waste water, commercial activated carbon (AC) is widely used as an adsorbent, showing excellent adsorption capacity due to its large specific surface area and abundant active adsorption sites on the surface (Ihsanullah et al., 2016; Kołodyńska et al., 2017; Rao & Kashifuddin, 2016; Zhou et al., 2015).

However, high cost, its non-renewable production source, and difficulties in regeneration have encouraged researchers to find alternative cost-effective adsorbents. To resolve the issues, biochar derived from various biomass has been proposed as an economical alternative for the removal of heavy metals from wastewater. Biochar is a black carbon produced during the pyrolysis of biomass under oxygen-limited conditions and at a relatively low temperature below 700 °C. Waste biomass which includes crop residues, forestry waste, animal manure, food processing waste, paper mill waste, municipal solid waste, and sewage sludge has been widely used in biochar production (Cantrell et al., 2012; Ahmad et al., 2014). Recently, biochar as an environment-friendly replacement for AC and adsorbents of other carbon materials has been proposed (Cha et al., 2016).

Specific surface areas for biochar range from less than 10 to 400 m2/g for pyrolysis temperatures below 400 °C and 550 – 600 °C, respectively (Brown, 2009). Biochar has generated renewed interests due to the abundance of resources for raw materials, its high porosity, its surface functional groups, and its high treatment efficiency on various pollutants (Abdul et al., 2017; Kanjanarong et al., 2017). Furthermore, biochar has successfully been used to treat wastewater and contaminated soils (Fahmi et al , 2018). It has been acknowledged that the use of biochar as a biosorbent for the treatment of wastewater and soil contaminated with heavy metals may provide a better alternative to conventional high-cost sorbents such as AC (Sohi, 2012).

Yet, the efficiency of jackfruit-extracted biochar as an adsorbent has not been examined in-depth. A comprehensive literature survey revealed that not many studies have been performed on the utilization of biomass-based biochar adsorbents although it is well known that traditional AC is more expensive and less environment-friendly compared to the biochar (Cha et al., 2016). A few investigations on the biomass-based biochar indicated that it can drastically reduce the overall cost of wastewater treatment against heavy metals (Kanjanarong et al., 2017). Current study focuses on the utilization of jackfruit waste-based biochar due to its two advantages. First, it helps to clean the environment through the utilization of agricultural waste material into useful material. Second, jackfruit-based biochar is low cost, plentiful, and an environment-friendly material for the removal of heavy metals from wastewater. According to the data reported by the Ministry of Agricultural and Agro-Based Industry of Malaysia, the annual production of jackfruit in 2011 was projected at 56,631 MT, translating to approximately 33,979 MT of jackfruit peels as byproducts. Retrieved from (MAAIM, 2011).

These notable benefits make jackfruit-based biochar unique and sustainable for water treatment against heavy metal contamination. Similar to other biochar, jackfruit-based biochar has excellent capacity to remove several contaminants from aqueous solutions, and thus, may constitute a basis for an untapped technology towards drinking water treatment. As the novelty in this study, waste agricultural biomass, especially the jackfruit waste peel and seeds were used to produce the biochar for the removal of heavy metals from water. Very few reports have revealed the use of jackfruit waste as an adsorbent (Inbaraj & Sulochana, 2004; Prahas et al., 2008; Rosli et al., 2015; Premachandra et al., 2017). In order to tap into the sustainable potential of jackfruit waste-based biochar as an effective adsorbent, this study explored a method to treat wastewater contaminated with heavy metals for lessening the threats of toxicity to human health. Based on these research gaps, the following objectives are set.

1.3 Objectives of the Study

The main objective of this study is to synthesize biochar from jackfruit waste and evaluate its efficiency on the adsorbent of heavy metals. To achieve this aim, the following objectives should be accomplished.

i. To synthesize activated biochar from jackfruit waste (*Artocarpus heterophyllus Lam*) using chemical activation technique.

ii. To evaluate the physiochemical properties of jackfruit-based Biochar using various characterization techniques.

iii. To study the optimum conditions (pH, temperature, time, initial concentration and biochar dosage) on the removal of heavy metals from contaminated water.

1.4 Research Questions

2. what is the best method/technique which can be used to synthesize Biochar from Jackfruit waste seeds and peel?

3. which are the crucial Physico-chemical properties of the synthesized biochar?

4. What are the optimum conditions for the efficient removal of heavy metals from the polluted water?

1.5 Scope of the Study

To achieve the proposed research objectives, the scope is defined as follows:

i. Synthesis of activated biochar from jackfruit waste of peel and seed and subsequent evaluation of their heavy metal removal efficacy.

ii. Activation of the biochar through phosphoric acid via pyrolysis at 500 °C to achieve carbonization to enhance its heavy metal removal efficiency.

iii. Characterization of the synthesized biochar according to their physical and chemical properties using diverse analytical techniques such as field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) spectroscopy, Fourier transformed infrared (FTIR) spectroscopy, X-ray diffraction (XRD) analysis, thermogravimetric analysis (TGA), CHNS, Raman spectroscopy, particle size, and BET of activated biochar.

iv. Evaluation of the efficiency optimization of the proposed jackfruit extracted biochar under different conditions including varying pH, temperatures, contact times, and concentrations of adsorbent in treating contaminated water.

v. Determination of adsorption process by biochar following the Langmuir and Freundlich isotherms in terms of the interaction between solutes and adsorbent.

vi. Determination of the adsorption kinetics to describe the solute uptake rate, which in turn presides over the adsorption contact time through the processes of first and second order reaction.

1.6 Significance of the Study

Disposal of agricultural waste has been identified as one of the global major problems. This challenge can be handled by transforming agricultural waste to adsorbents for removing heavy metals from water. In developing countries, various human activities related to mining, industrialization, and urbanization as well as rapid population growth are responsible for the gradual water contamination along rivers and other stagnant water bodies. Therefore, constant efforts have been made to device a sustainable way of mitigating the deadly heavy metal pollution of water. Heavy metals when ingested at high concentration beyond recommended limit can cause various degrees of diseases and illnesses to human beings that may eventually lead to death. It is important to find sustainable ways of treating industrial waste water before entering main rivers and subsequently main water bodies. Various studies have been carried out around the world, utilizing different techniques for the removal of heavy metals from contaminated water. However, these techniques are usually expensive and less environment-friendly. This study adopted adsorption technique using agricultural waste-based adsorbent. The adsorption technique has been widely used in recent times for heavy metal removal from water. Therefore, this concept is not new but the research is important as it provides an innovative approach in terms of utilizing the jackfruit seed and peel as adsorbent in waste water treatment against poisonous heavy metals. This process is economically feasible, technically simple, and environmentally benign. Jackfruit waste has no economic value and often creates a serious disposal problem in local environment. Thus, utilizing jackfruit waste as an alternative and low-cost adsorbent would increase its economic value, help reduce the cost of waste disposal, and considerably reduce the problem of environmental pollution. Transforming abundant agricultural waste products into materials such as biochar that can absorb contaminants can have environmental implications for improving waste management and protecting the environment. Thus, the present study aims to investigate the potential of the abundantly available jackfruit waste as an adsorbent in the removal of heavy metal toxicity from aqueous solutions.

1.7. Conceptual Framework of the Study

To accomplish the proposed objectives the following research framework was conceptualized:

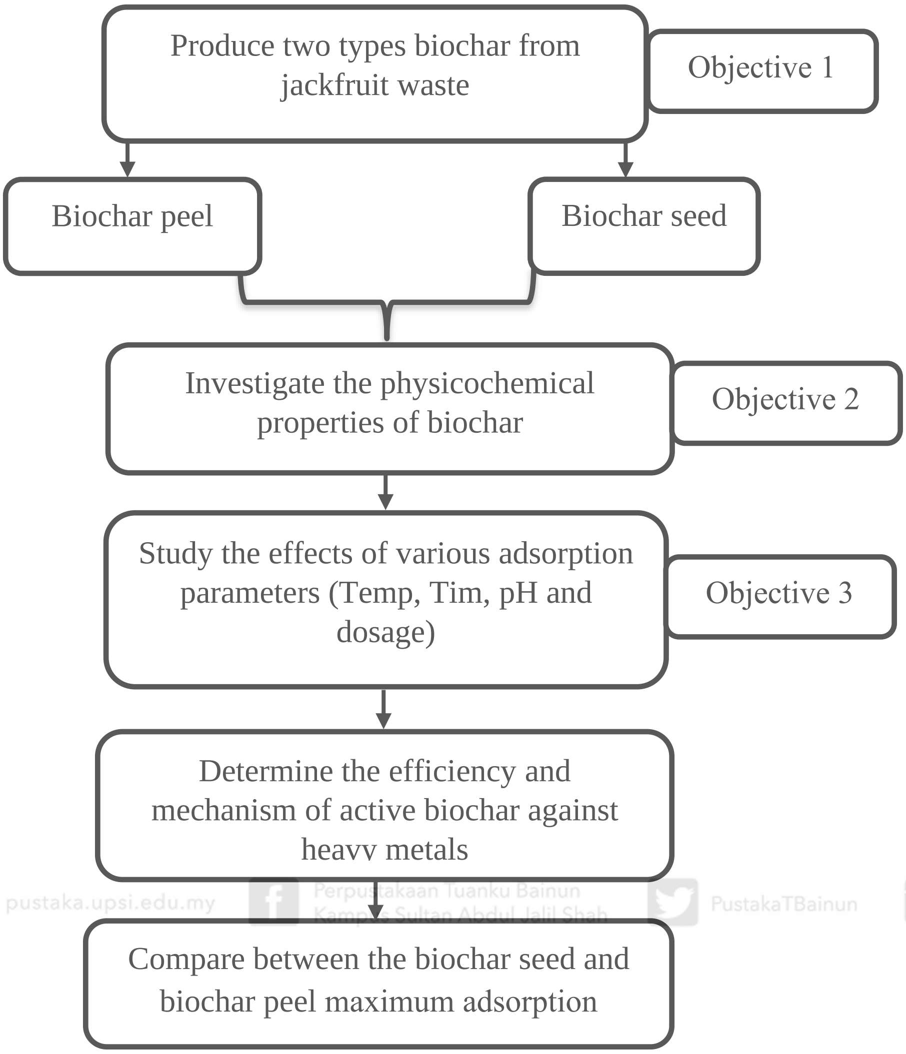

Figure 1.1. Conceptual Framework of The Study

1.8 Limitations of the Study

Inadequacy of previous literature specifically on jackfruit waste is the major limitation of this research. Only a few studies have been carried out on the biochar adsorbent removal of heavy metals from wastewater. Nevertheless, information is available on other adsorption techniques using various adsorbents for the water treatment of heavy metal contamination. These provided general secondary information needed for the research work. The purpose of this study was to remove selected heavy metals, namely

Cd (II), Cu (II), Pb (II), Fe (II) and Mn (II) from aqueous solutions and to evaluate the influence of activated biochar prepared.

1.9 Thesis Organization

This thesis consists of six chapters. Chapter 1 contains the introduction with a brief background to highlight the significance and necessity of this research. It emphasizes the research problems, objectives, scope, and significance of the undertaken study. Chapter 2 provides a comprehensive and critical overview of the relevant literature. It includes the basics on environmental pollution; causes of environmental pollution; water quality and treatment methods; sources, toxicity, and classification of heavy metals; sorption relationship between charcoal, activated carbon, and biochar; and adsorption and its advantages over the techniques used. Chapter 3 describes the research methodology of the biochar synthesis and characterizations using various analytical techniques to determine its physicochemical properties. Various theoretical formalisms and thermodynamic models to determine the heavy metal removal efficiency of the prepared activated biochar are also explained. Chapter 4 includes all the experimental results, analyses, interpretations, comparisons, validation, and theoretical descriptions to understand the kinetic mechanisms and thermodynamics of the proposed biochar. Chapter 5 outlines the discussion on the results presented in Chapter 4, wherein all the attributes and explanations in terms of various physicochemical mechanisms are highlighted. Chapter 6 concludes the thesis with some recommendations for future research.