# REMOVAL OF LEAD, CADMIUM, AND NICKEL IONS FROM AQUEOUS SOLUTION USING OKRA AND CHILLI SEEDS AS ADSORBENT

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**by**

**HANG LIPO ANGELINA A/P AYOB**

**Dissertation submitted in partial fulfilment of the requirements for the degree of Bachelor of Science in Forensic Science**

**February 2025**

# CERTIFICATE

This is to certify that the dissertation entitled

**"REMOVAL OF LEAD, CADMIUM, AND NICKEL IONS FROM AQUEOUS SOLUTION USING OKRA AND CHILLI SEEDS AS ADSORBENT"**

is the bona fide record of research work done by

HANG LIPO ANGELINA A/P AYOB

During the period of October 2024 to February 2025 under my supervision. I have read this dissertation and, in my opinion, it conforms to acceptable standards of scholarly presentation. It is fully adequate in scope and quality to be submitted in partial fulfilment for the degree of Bachelor of Science in Forensic Science.

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**DECLARATION**

I hereby declare that this dissertation is entirely my work, and has not been previously submitted, in whole or in part, for any degree or academic qualification at Universiti Sains Malaysia, or any other institution. All sources of information, ideas and reference used in this research have been properly acknowledged. I grant Universiti Sains Malaysia to store, reproduce, and distribute this thesis, in full or in part, for academic and research purposes, under the university's policies and regulations.

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(HANG LIPO ANGELINA A/P AYOB)

Date: 27/2/2025

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## LIST OF SYMBOLS AND ABBREVIATIONS

| | |
|---|---|
| % | Percentage |
| ˚ | Degree (angle or curvature) |
| ˚C | Degree Celcius |
| + | Plus |
| - | Minus |
| × | Multiply |
| = | Equal |
| [M] | Concentration of heavy metals |
| ATR-FTIR | Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy |
| BET | Brunauer–Emmett–Teller Surface Area Analysis |
| C=O | Carbonyl group |
| C-H | Carbon-hydrogen bond |
| C-O | Carbon-oxygen single bond |
| COOH | Carboxyl group |
| Cd | Cadmium |
| $cm^{-1}$ | Wavenumber (used in spectroscopy) |
| DNA | Deoxyribonucleic Acid |
| et al. | And others |
| FAAS | Flame Atomic Absorption Spectroscopy |
| FAO/WHO | Food and Agriculture Organization / World Health Organization |
| g | Gram |
| HCl | Hydrochloric Acid |
| $HNO_3$ | Nitric Acid |
| $H_2SO_4$ | Sulfuric Acid |
| L | Liter |
| mL | Mililiter |
| mg/kg | Miligrams per kilogram |
| mg/L | Miligrams per liter |
| mm | Milimeter |
| $NH_2$ | Amine group |
| Ni | Nickel |
| O-H | Hydroxyl group |
| pH | Measure of acidity or alkalinity |
| Pb | Lead |
| ppm | Parts per million |
| $R^2$ | Coefficient of determination |
| SEM | Scanning Electron Microscopy |
| µg/L | Micrograms per liter |

## LIST OF APPENDICES

**PENYINGKIRAN ION PLUMBUM, KADMIUM DAN NIKEL DARIPADA LARUTAN AKUEUS MENGGUNAKAN BIJI SAYUR BENDI DAN CILI SEBAGAI PENJERAP**

## ABSTRAK

Pencemaran logam berat dalam sumber air, terutamanya plumbum (Pb), kadmium (Cd), dan nikel (Ni), menimbulkan ancaman besar terhadap kesihatan manusia disebabkan oleh ketoksikannya, sifatnya yang sukar terurai dalam persekitaran, serta keupayaan bioakumulatifnya. Kajian ini menilai keberkesanan biji okra (*Abelmoschus esculentus*) dan biji cili (*Capsicum annuum L.*) sebagai bahan penjerap untuk menyingkirkan logam berat ini daripada larutan akueus. Beberapa parameter telah dikaji dalam eksperimen penjerapan ini, termasuk kepekatan awal, dos penjerap, masa sentuhan, dan suhu. Spektroskopi Serapan Atom Nyalaan (FAAS) digunakan untuk mengukur kepekatan ion Pb, Cd, dan Ni. Manakala, Spektroskopi Inframerah Transformasi Fourier (FTIR) digunakan untuk mencirikan biosorben sebelum dan selepas rawatan bagi mengenal pasti kumpulan berfungsi yang terlibat dalam proses penjerapan. Parameter optimum bagi penyingkiran Pb, Cd, dan Ni menggunakan biji okra adalah 1 ppm kepekatan awal, 0.5 g dos penjerap, 20 minit masa sentuhan, dan suhu 30°C, dengan kecekapan penyingkiran masing-masing sebanyak 84.9% (Pb), 87.3% (Cd), dan 28% (Ni). Bagi biji cili, dengan keadaan yang sama tetapi dengan menggunakan 1.0 g dos penjerap, kecekapan penyingkiran yang diperoleh adalah 92% untuk Pb, 82.4% untuk Cd, dan 37.4% untuk Ni. Berdasarkan pemerhatian, Pb menunjukkan peratusan kecekapan penyingkiran tertinggi berbanding Cd dan Ni, manakala Ni mencatatkan peratusan terendah bagi kedua-dua jenis penjerap. Walau bagaimanapun, dapatan kajian ini menunjukkan bahawa biji okra dan biji cili

berpotensi sebagai bahan penjerap semula jadi bagi menyingkirkan logam berat Pb, Cd, dan Ni daripada larutan akueus.

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## ABSTRACT

Heavy metal contamination in water sources, especially lead (Pb), cadmium (Cd), and nickel (Ni), poses a significant threat to human health, due to their toxicity, persistence in the environment, and bioaccumulative nature. This study examined the effectiveness of okra (*Abelmoschus esculentus*), and chilli (*Capsicum annuum L*) seeds as adsorbents for these metals removal from aqueous solution. Several parameters, including initial concentration, adsorbent dosage, contact time, and temperature, were conducted for this adsorption experiment. Flame Atomic Absorption Spectroscopy (FAAS) was used to measure Pb, Cd, and Ni ion concentrations. Fourier Transform Infrared Spectroscopy (FTIR) was used to characterise biosorbents before and after treatment to discover functional groups involved in the adsorption process. The optimal parameters for Pb, Cd, and Ni removal using okra seeds were 1 ppm of initial concentration, 0.5 g adsorbent dosage, 20 minutes of contact time, and 30°C temperature, resulting in removal efficiencies of 84.9%, 87.3%, and 28%, respectively. For chili seeds, the same conditions with a 1.0 g adsorbent dosage resulted in 92% removal efficiency for Pb, 82.4% for Cd, and 37.4% for Ni. Based on the observations, Pb yielded the highest percentage removal efficiency compared to Cd and Ni, whereas Ni had the lowest for both adsorbent types. However, the findings indicated that both okra and chilli seeds have the potential to remove Pb, Cd, and Ni heavy metals from aqueous solution.

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Water contamination from heavy metals such as lead (Pb), cadmium (Cd), and nickel (Ni) has become a major global concern due to their toxicity, persistence, and possible bioaccumulation. These heavy metals most likely come from industrial, agricultural, and residential activities, contaminating aquatic habitats and causing substantial health hazards even at low levels (Kumar Yadav et al., 2018). These contaminants enter water systems via a variety of channels, including industrial waste disposal, mining activities, agricultural runoff, and atmospheric deposition, accumulating hazardous metals in lakes, rivers, and groundwater sources (Genchi et al., 2020).

Exposure to these heavy metals has severe effects on health, such as affecting the central nervous system and fetal development, causing cancer and kidney damage through contaminated food or water, and also causing respiratory problems and allergic reactions with prolonged exposure (Maftouh et al., 2023).

Heavy metals have been removed from wastewater using a variety of procedures, including oxidation, precipitation, membrane filtration, and ion exchange; however, these traditional technologies are generally associated with significant operational expenses, energy consumption, and the production of secondary pollutants (Raji et al., 2023). Adsorption has evolved as an effective and sustainable option due to its low cost, high efficiency, and capacity to remove metals at trace levels (Maftouh et al., 2023).

Recent studies have focused on agricultural waste as potential biosorbents, however, few studies have looked at the usage of okra and chili seeds for heavy metal removal. This study evaluates the adsorption ability of okra and chilli seeds for the removal of $Pb^{2+}$, $Cd^{2+}$, and $Ni^{2+}$ from aqueous solutions. Furthermore, it evaluates how changes in functional groups on an adsorbent surface, affect the adsorption process by analyzing the adsorbent material's properties before and after binding to metal ions, typically using techniques such as Fourier Transform Infrared Spectroscopy (FTIR) to identify specific functional groups involved in the binding process.

## 1.2 Problem Statement

The toxicity, persistence, and potential bioaccumulation of heavy metals such as lead (Pb), cadmium (Cd), and nickel (Ni) in water solutions poses significant risks to the environment and health. These contaminants have negative impact on aquatic ecosystems and also human health, which can cause various illnesses such as neurological disorders, cancer, and kidney damage.

Effective removal of these pollutants is consequently critical. While there are several heavy metal remediation technologies available, such as chemical precipitation, ion exchange, and membrane filtration (Fu & Wang, 2011), agricultural waste's potential as cost-effective and environmentally acceptable biosorbents has yet to be explored. Existing technologies can be costly, energy-intensive, and produce secondary waste, limiting their usefulness, especially in resource-constrained environments.

Considering their abundance and capacity for metal binding, okra (Abelmoschus esculentus) and chili pepper (Capsicum annuum) seeds, which are readily available agricultural byproducts, offer a possible alternative (Kwikima et al., 2021).

This study aimed to fill this gap by quantifying the adsorption capacity of okra and chili seeds for removing Pb, Cd, and Ni from aqueous solutions under varying conditions, characterising changes in the functional groups of these seeds before and after metal binding using techniques such as ATR-FTIR, and assessing the potential of these biosorbents for practical use in wastewater treatment.

## 1.3 Objectives of the Study

### 1.3.1 General Objective

To remove Pb, Cd and Ni ions from aqueous solution using okra and chilli seeds as adsorbent.

### 1.3.2 Specific Objective

1. To characterise okra and chilli seeds before and after removal of Pb, Cd, and Ni ions from aqueous solution using Fourier Transform Infrared Spectroscopy (FTIR).
2. To perform the removal process of Pb, Cd, and Ni ions from aqueous solutions using okra and chilli seeds, analysed through Flame Atomic Absorption Spectroscopy (FAAS).
3. To investigate the effectiveness of adsorption process using okra seeds and chilli seeds as natural waste adsorbents towards the removal of Pb, Cd, and Ni from aqueous solution using different parameters such as

initial concentration of metal ions solutions, adsorbent dosage, contact time and temperature.

## 1.4 Significance of the study

This study is significant because it introduced and explored the use of okra and chili seeds as potential low-cost and ecologically friendly adsorbents for the removal of lead, cadmium, and nickel from aqueous solutions. The increasing contamination of water sources with toxic metals poses significant environmental and health problems, necessitating the development of long-term cleanup techniques.

This work proposed an alternative, cost-effective option for water treatment by employing okra and chilli seeds as adsorbents, which is especially useful in resource-constrained areas. Previous studies have shown that agro-based adsorbents can remove heavy metals with more than 90% efficiency, making this technique both effective and sustainable (Aftab et al., 2024). Additionally, agricultural waste-derived adsorbents help to conserve the environment by recycling organic waste that would otherwise pollute. The successful application of this strategy has the potential to improve water quality, limit human exposure to hazardous metals, and encourage safer agricultural practices (Alsulaili et al., 2023).

In addition to environmental and health benefits, this study has forensic applications in environmental monitoring, public health risk assessment, and food safety enforcement. The use of Flame Atomic Absorption Spectroscopy (AAS) in this study provides a significant tool for forensic investigations involving heavy metal pollution. In environmental forensics, FAAS can be used to determine the efficacy of bioremediation procedures by detecting and quantifying heavy metal buildup in contaminated soils and water bodies. For example, chilli seeds exposed to polluted environments may collect heavy metals, and their analysis by FAAS can give critical evidence in identifying pollution sources and developing remediation strategies (Ali et al. 2013).

This forensic technique aids in pollution detection and regulatory actions for damaged agricultural fields. Furthermore, the study advances forensic toxicology by evaluating the potential health concerns linked with heavy metal contamination in dietary sources. Toxic metals like cadmium and lead are especially dangerous to vulnerable populations, such as youngsters and pregnant women. FAAS may identify high quantities of heavy metals in agricultural items like chilli seeds, which could suggest contamination in the food supply chain. Identifying such pollutants is critical for public health risk assessments since prolonged exposure to toxic metals can cause neurological damage, organ failure, and other serious health consequences.

By incorporating forensic toxicological analysis, this study helps to identify and mitigate dietary exposure to harmful compounds, improving food safety monitoring. Additionally, this study has forensic implications in regulatory compliance and legal enforcement of food safety regulations. Heavy metal contamination in food products is strictly regulated, and meeting legally acceptable levels is critical to protecting public health.

This study used FAAS to provide a scientific basis for determining whether agricultural goods fulfill established safety requirements. The findings can be used by regulatory organizations to limit the distribution of contaminated food and impose stronger quality control standards. Evidence-based methods for food safety compliance lower the risk of heavy metal exposure, which result in safer agricultural practices and consumer protection.

In conclusion, this study contributed to environmental sustainability and water purification while playing a vital role in forensic science. Bioremediation is combined with forensic environmental monitoring, toxicological assessment, and regulatory enforcement to provide a multidisciplinary approach for heavy metal contamination. The findings lay the foundation for better water treatment, stronger public health initiatives, and stricter food safety rules, all of which contribute to a cleaner, safer world.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Heavy Metal in Kelantan River Water

The Kelantan River is the main river located in the state of Kelantan. The reservoir covers about 11,900 km² in northeastern Malaysia, including part of the National Park, and drains into the South China Sea. As the main river in the state of Kelantan, it plays an important role in maintaining the population of about 0.5 million people in the Kelantan basin, therefore there is a lot of industrial development such as mining, agriculture, plantation irrigation, small-scale fishing industry, and sand (Hee et al., 2019). This contributes to heavy metal pollution, which is mostly found in the form of particles in the Kelantan River. This metal contamination indicates an increase in suspended solids, which leads to a higher amount of particulate metals. This demonstrates how metals contribute significantly to the water column. Studies show that this is significantly linked to the activities mentioned above.

Significant Pb levels were found in the densely populated capital city, Tanah Merah, and Kuala Krai urban districts, indicating that urban runoff, sewage effluent, oil spillage, and boating activities all contribute to higher Pb concentrations in the environment (Pooveneswary et al., 2019). In addition, Ni levels were found to be higher along the Lebir River, at its confluence, in the capital city, and in remote coastal places.

However, the relatively stable Ni levels along the river indicated that the majority of the Ni came from lithogenic sources. However, the widespread use of agricultural chemicals, building materials, and paints near metropolitan centers may aggravate Ni levels, particularly if heavy metals are not continuously and regularly monitored (Pooveneswary et al., 2019).

Subsequently, Cd was discovered along the Galas River, indicating a minor lithogenic input from natural erosion or weathering. Similar to Ni, higher Cd levels were also detected near the Lebir River and in the capital city, it had been concluded that proximal boating, mining, smelting, and recycling of metals or ores may have contributed to the increase in Cd levels (Pooveneswary et al., 2019).

The average amounts of heavy metals (in descending order) in the Kelantan River descended in the order of Mn > Zn > Pb > Cr > Cu > As > Ni > Cd, and Mn > Pb > Zn > Cr > As > Cu > Ni > Cd for the nearshore surface sediments (Hee et al., 2019). Concentration Seasonal fluctuation reveals a distinct peak of particulate metals, such as Zn, Fe, and Pb, during the dry season.

The natural phenomena are thought to be influenced by lower river flow rates, which may have diminished dilution effects and hence reveal more particulate accumulation of environment Water Quality Index (DOE-WQI). This necessitates the monitoring and management of anthropogenic activities to limit their impact on the river's environment.

## 2.2 Exposure Pathway of Lead, Cadmium, and Nickel to Humans and Living Organism

This study used FAAS to provide a scientific basis for determining whether agricultural goods fulfill established safety requirements. The findings can be used by regulatory organizations to limit the distribution of contaminated food and impose stronger quality control standards. Evidence-based methods for food safety compliance lower the risk of heavy metal exposure, which result in safer agricultural practices and consumer protection.

Heavy metal exposure has shown to be a significant concern to human health, mostly due to their propensity to cause membrane and DNA damage, as well as disrupt protein function and enzyme performance. These metals disrupt native protein functioning by attaching to free thiols or other functional groups, accelerating the oxidation of amino acid side chains, altering protein folding, and/or displacing critical metal ions in enzymes (Witkowska et al., 2021).

An exposure pathway is a route of how a person comes into contact with heavy metals. There are three main exposure pathways which are consumption, ingestion, and direct touch (Witkowska et al., 2021). First, lead (Pb) can enter the body primarily by eating and inhalation, both of which are hazardous to health. Ingesting involves consuming contaminated materials such as water, food, or soil particles. For example, ingestion can occur in young children by placing lead-containing objects or soil into their mouths or through inhaling small particles from lead-containing dust. Especially for aquatic ecosystems, this is because it can accumulate in sediments and bioaccumulate in primary producers and consumers, then forming food webs linked to the exposure of higher trophic levels to increased levels of lead (Raj & Das, 2023). As a result, this bioaccumulation can cause increased amounts of lead in fish and other aquatic organisms, which, then can also lead to poisoning when consumed by humans.

Additionally, cadmium is mostly consumed through food, which is grown in plants, fish, and other species, it can quickly pass from soil to plant. For example, rice grown in cadmium-contaminated water may generate elevated levels of cadmium in the rice, which can endanger the health of the consumers. Furthermore, cadmium is also an inhalation hazard to humans, with sources ranging from industrial operations to released aerosol from cigarette smoke. According to the World Health Organization (n.d.), people working in the metal smelting and refining industries, as well as those involved in the manufacture of batteries, mechanical plating, and fossil fuel combustion are more prone to inhaling cadmium dust or fumes, exposing them to high levels of this metal.

Finally, nickel exposure can occur through the consumption of contaminated water, food, or soil, since it enters the environment via industrial discharges. For example, humans consume crops irrigated with water with a high proportion of nickel. Furthermore, aquatic creatures can absorb nickel directly into their bodies through water via their gills or skin, resulting in bioaccumulation throughout the food chain. Some species may come into contact with polluted soil, and nickel aerosols may enter the body via dust inhalation or breath (Suljević et al., 2023).

## 2.3 Overview of Adsorption

The term adsorption was first introduced in 1881 by Heinrich Kayser, a German scientist at the time, and is still used today. It is a process or phenomenon that occurs when atoms, ions, or molecules adhere to a surface from a gas, liquid, or dissolved solid. According to (Chen et al., 2022) adsorption is the mass transfer of chemicals from the liquid phase to a solid surface, which separates them from watery media. The substances known as adsorbates are those that have been adsorbed, and the adsorbing phase is adsorbents.

In contrast, absorption is a process in which atoms, molecules, or ions enter a bulk phase, such as liquid or solid material. Molecules undergoing absorption are taken up by the volume rather than the surface (as with adsorption). The absorption process is when something takes in another substance. The absorption process involves a material capturing and transforming energy. Another distinction between absorption and adsorption is that the absorbent distributes the substance it collects throughout the entire, whereas the adsorbent distributes it through the surface. Absorption is commonly defined as any gas or liquid penetrating the adsorbent.

### 2.3.1 Mechanism of Adsorption and Application

Adsorption occurs when a gaseous, liquid, or solid substance (substrate) adheres to the surface of a solid or liquid (sorbent). There are several adsorption systems, including liquid-gas and liquid-liquid. If a liquid material is an adsorbent, it forms an interfacial layer known as a film, micelle, or emulsion. The interfacial layer model is the approved adsorption mechanism for solid-liquid or solid-gas systems with a solid adsorbent. This study focuses on the solid-liquid system, with the interfacial layer representing the equilibrium between the adsorbent and bulk phases. This system involves the binding of the substrate to the sorbent surface, followed by the sorbent's surface layer.

There are two explanations for the interfacial layer mechanism chemical adsorption (chemisorption) and physical adsorption (physisorption). Chemical adsorption is a mechanism that occurs between substrate, and adsorbent through electron density rearrangement, which results in ionic or covalent bonds. The chemical connection between the adsorbent and adsorbate causes the enthalpy of chemisorption to balance between 200 and 400 kJ/mol. Chemisorption is intimately related to surface area and temperature, influencing adsorption efficiency.

In contrast, physical adsorption (physisorption) typically occurs in weak contacts, such as weak van der Waals forces. As a result, neither the substrate nor the sorbent's chemical structure changed (Alaqarbeh, 2021). It is an exothermic reaction with a lower enthalpy of adsorption, ranging between 20 and 40 kJ/mol. Physisorption, unlike chemisorption, is non-specific in the sense that the adsorbate can be adsorbed to