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EFFECT OF THE INITIAL CONCENTRATION METAL ION AND ADSORBENT DOSE ON HEAVY METAL REMOVAL USING NAPIER GRASS

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DECLARATION

I declare that this thesis entitled "Effect of the Initial Concentration Metal Ion and Adsorbent Dose on Heavy Metal Removal Using Napier Grass" is the results of my own research except as cited in the references.

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Effect of the Initial Concentration Metal Ion and Adsorbent Dose on Heavy Metal Removal Using Napier Grass

ABSTRACT

In recent decades, the release of heavy metals into aquatic ecosystems has raised concerns in Malaysia. These pollutants primarily enter water bodies due to various industrial activities, which gained momentum with the initiation of a developmental plan. The identified pollutants encompass lead, chromium, mercury, uranium, selenium, zinc, arsenic, cadmium, gold, silver, copper, and nickel. These hazardous substances can originate from mining operations, ore refining, sludge disposal, incinerator fly ash, processing of radioactive materials, metal plating, and the manufacturing of electrical equipment, paints, alloys, batteries, pesticides, or preservatives. Over the past few decades, various methods have been developed for the treatment and removal of heavy metals include adsorption methods. Extensive research has been conducted to remove heavy metals from industrial wastewater using adsorbents produced from agricultural waste, often known as biosorbents. These bio-sorbents have proved safety and biodegradability, making them appropriate for the treatment of heavy metal-contaminated wastewater. Furthermore, Napier grass is a low-cost bio-sorbent capable of removing copper or lead from wastewater. Napier grass, which is mostly composed of cellulose, pectin, and lignin, is good at binding copper and lead ions, allowing them to be separated from wastewater. The biosorption of lead(Pb) from dilute aqueous solution using Napier grass as the biosorbent has been studied. The biosorption study was carried out as a function of initial metal ion concentration, and adsorbent dose. The residual Pb in solution were determined using atomic absorption spectrophotometer (AAS). The synthesized biosorbents were characterized by surface area analyzer. Effects of initial concentration heavy metal ion, and adsorbent dosage on the Pb(II) removal process had been optimized. The study on initial metal ion concentration showed that the biosorption was initial concentration dependent as maximum biosorption of Pb was obtained in the range 5-7 ppm. The biosorption was also adsorbent dose dependent as maximum biosorption was obtained within 0.5g-1.5g of the biosorbent. For the metal ions, the biosorption efficiency increased with increase in initial metal ion concentration.

Keywords : Napier grass, heavy metals, adsorption, removal, atomic absorption spectroscopy.

Kesan Kepekatan Awal Ion Logam dan Dos Penjerap terhadap Penyingkiran Logam Berat menggunakan Rumput Napier

ABSTRAK

Dalam beberapa dekad terkini, pelepasan logam berat ke dalam ekosistem akuatik telah menimbulkan kebimbangan di Malaysia. Pencemaran ini utamanya memasuki badan air akibat pelbagai aktiviti perindustrian, yang mendapat daya tarikan oleh pelaksanaan pelan pembangunan. Pencemaran yang dikenal pasti merangkumi plumbum, kromium, merkuri, uranium, selenium, zink, arsenik, kadmium, emas, perak, kuprum, dan nikel. Bahan-bahan berbahaya ini boleh berasal dari operasi penambangan, pemurnian bijih, pembuangan lumpur, abu terbang pembakaran, pemprosesan bahan radioaktif, pelat logam, dan pembuatan peralatan elektrik, cat, aloi, bateri, racun serangga, atau pengawet. Selama beberapa dekad kebelakangan ini, pelbagai kaedah telah dibangunkan untuk rawatan dan penyingkiran logam berat termasuk kaedah penjerapan. Kajian yang meluas telah dijalankan untuk mengeluarkan logam berat dari air sisa industri dengan menggunakan penjerap yang dihasilkan daripada sisa pertanian, yang sering dikenali sebagai biosorben. Biosorben ini terbukti selamat dan boleh diuraikan, menjadikannya sesuai untuk rawatan air sisa yang tercemar oleh logam berat. Selain itu, rumput Napier merupakan biosorben kos rendah yang mampu mengeluarkan kuprum atau plumbum dari air sisa. Rumput Napier, yang terutamanya terdiri daripada selulosa, pektin, dan lignin, terbukti berkesan mengikat ion kuprum dan plumbum, membolehkan pemisahan daripada air sisa. Kajian biosorpsi plumbum (Pb) daripada larutan akueus cair menggunakan rumput Napier sebagai biosorben telah dijalankan. Kajian biosorpsi dilakukan sebagai fungsi kepekatan ion logam awal dan dos penjerap. Pb terbaki dalam larutan ditentukan menggunakan spektrofotometer serapan atom (AAS). Biosorben yang disintesis dikarakteristikkan oleh pengukur kawasan permukaan. Kesan kepekatan awal ion logam berat dan dos penjerap pada proses penyingkiran Pb(II) telah dioptimumkan. Kajian ke atas kepekatan awal ion logam menunjukkan bahawa biosorpsi adalah bergantung kepada kepekatan awal kerana biosorpsi maksimum Pb diperoleh dalam julat 5-7 ppm. Biosorpsi juga bergantung kepada dos penjerap kerana biosorpsi maksimum diperoleh dalam julat 0.5g-1.5g biosorben. Bagi ion logam, keberkesanan biosorpsi meningkat dengan peningkatan kepekatan awal ion logam.

Kata Kunci: Rumput Napier, logam berat, penjerapan, penyingkiran, spektroskopi serapan atom.

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LIST OF ABBREVIATIONS

FTIR	Fourier Transform Infra-red
AAS	Absorption Spectrometer
SEM	Scanning Electron Microscope
DM	Dry matter
CP	Crude protein
ADL	Acid detergent lignin
ADF	Acid detergent fibre
NG	Napier grass
rNG	Raw Napier grass

LIST OF SYMBOLS

Rpm	Rotation per minute
cm	Centimetre
m	Metre
%	Percentage
℃	Celcius
µm	Micrometre
g	Gram
L	Litre
mL	Mililitre
ppm	Part per million
Å	Angstroms

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Napier grass or scientific name known as Pennisetum purpureum is a fast-growing perennial grass native to the world's tropical and subtropical climates. It is a versatile forage crop that is mostly used to feed cattle in cut and carry systems. Characterization and diversity studies on a small collection of Napier grasses revealed a moderate level of genetic variety and emphasized the availability of several desirable agronomic features as a fodder crop, particularly high biomass output (Alemayehu et al., 2017).

By raising the biomass yield of Napier grass by lowering the likelihood the invasiveness, the crop values as s source of bioenergy could be increased. However, as the cutting interval grew, the percentage of crude protein and metabolized energy significantly dropped. It is evident from the results that cutting a stand of Napier grass at 35 days will result in a higher yield and nutrient content. Harrison conducted digestion trials with fresh Napier grass and found the digestion coefficients to be: for crude protein 63, for crude fiber 64, for nitrogen-free extract 60, and for crude fat 57 (Harrison E., 1942).

The appearance for the rhizomatous, tufted, and strong root system of Napier grass grows from the nodes of its creeping stolons. The culm is rough and can range in height from 4 to 7 metres. The plant can grow in clumps that are up to 1 m broad and extremely dense. The leaves are long, wide, and bluish-green in colour. Furthermore, the leaves should flat, linear, and hairy at the base. The inflorescence is a yellow-brown to purplish, rigid, terminal bristly spike that is 15 cm to 20 cm long. Around a hairy axis, spikelets are organized there contain little to no seed formation occurs. When seeds do exist, they are really tiny. Essential conditions in warm, tropical and subtropical climates, Napier grass grows best and thrives at elevations of up to 2000 metres above sea level.

The temperature range of 25 to 40 degrees celsius generally results in excellent performance. In fact, frozen conditions and water logging affect Napier grass quite negatively. So the approximately temperature range of 25 to 40 degrees celsius generally. There are rarely many full-form seeds produced when Napier grass is planted. Stem cuttings are therefore the primary method of proliferation such as in both the rows and between them, a few centimetre apart furrows are used to plant the cuttings with five internodes (Haegele & Arjharn, 2017). The widespread use of Napier grass for soil and water conservation in mountainous slope farming areas is one of its additional uses.

In previous researched, cultivation of agricultural crops and harvesting generate an abundant amount of waste that can be used for wastewater treatment. These agricultural wastes are primarily composed of cellulose, hemicelluloses, and lignin, but may also contain other functional groups such as hydroxyl, aldehyde, carbonyl, carboxyl, phenolic, and ether groups, which can interact with pollutants in wastewaters via a variety of selective binding mechanisms or interactions(Bajaj, n.d.).

1.2 Problem Statement

Environmental degradation is one of the most pressing concerns facing society today. It was affecting living species until it reached frightening proportions in recent years, and it is still increasing exponentially now. Heavy metal is harmful and is regarded as one of the types of pollution that has a direct impact on humans and animals. For example, certain industrial effluent may contain lead, copper, cadmium, or chromium, causing substantial groundwater pollution and potentially contaminating groundwater resources. On the health side, heavy metal is a toxic and affect the human health which consume drinking water which exposing to the chemicals and might damage the central nervous system, respiratory system, kidney and blood system.

Secondary pollutant might form caused by the bad water treatment of the industrial wastewater. Previous studied proof us the effective method and techniques to treated the industrial wastewater especially on the water contain heavy metal. Biosorption technique has the potential to be a long-term and cost-effective means of eliminating dyes from a number of sources. There are many conventional methods available for the purpose of the treatment of the ions from heavy metals from wastewater that is precipitation using chemical compound or membrane filtration. The unconventional procedure of heavy metal ion treatment can overcome the shortcomings of conventional approaches, and one such method is to employ biosorbent to adsorb the ions from heavy metal, which is both economical and environmentally. Biosorbent materials that are plentiful in nature, such as biomass, agricultural waste, algae, bacteria, industrial waste, fish scales, and many more, have been shown to be good adsorbents for the removal of heavy metal ions.

1.3 Objectives

The objective of this study are:

1. To evaluate the effect of initial heavy metal ion concentrations on efficiency of heavy metal adsorption by Napier grass.
2. To evaluate the effect of adsorbent dose on efficiency of heavy metal adsorption by Napier grass.
3. To evaluate the surface area of the raw Napier grass using Surface Area Analyzer.

1.4 Scope of Study

This study was designed to develop a better understanding on how to treat the water pollution that effects of the environmental using the Napier grass as a biosorbent without causing the secondary pollution undergo the adsorption process. In this study, the mature Napier grass stem was used as a raw material for the biomass processing necessary to create biosorbents and its ability to absorb heavy metals ion was assessed. The method of the treatment act physically through the adsorption of the heavy metal removal. Additional research in a variety of domains is required for biosorbents to reach their full potential. There are just a few of the techniques utilised to improve the sorption activity of biosorbents. Microbial biomasses, agricultural waste, industrial by products, or natural materials have all been hailed as promising substrates for metal removal by biosorption due to their high efficiency, low cost, and great abundance. These biological materials are suitable for biosorption processes. (Tongpoothorn et al., 2020)

The sample was undergo pretreatment process include drying and grinding into powder form. The results of the research conducted therefore were analysed based on the surface of the biosorbent after the pretreatment using the Surface Area Analyzer.(Shing et al., 2019a). Moreover, the quantitative analysis using Atomic Absorption Spectroscopy (AAS) to analyse the percentage removal lead ion before and after in the wastewater.

1.5 Significances of Study

The research of this study able to proof that Napier grass as a wild crop and agricultural waste known as high biomass production that can bind positively charged of the heavy metal ions to derived the Napier grass as the biosorbent. The study is important for produce the biosorbent for the wastewater treatment using the agricultural waste and eco-friendly material to avoid the secondary pollutant and low cost of the production.

Biosorbent using Napier grass for wastewater treatment an effective economically should be developed more by applied in each of the industrial to treat the wastewater eco-friendly. Therefore, the area surface of the biosorbent before applied in adsorption process must efficient and suitable for that type of biosorbent either in solid, powder of liquid form. (Raji et al., 2023)

Napier grass was adsorbed onto metal ions from aqueous solution in a batch adsorption study with a variety of settings. In basic solution, metal adsorption increased and reduced. This suggests that the heavy metal isn't very stable in basic solution. As a result, Napier grass can remove heavy metals at the initial concentration and dosage. Furthermore, Napier grass can be employed as an adsorbent for heavy metal removal if the maximum contact time, pH, and temperature are maintained(Qasem et al., 2021a).

In this investigation, the best ideal condition for the heavy metal removal were observed by the amount of the adsorbent dosage and the initial concentration controlled, small particle size of the adsorbent less than 300 microns, and a shaking speed of 200 rpm to make sure the adsorbent fully react with the adsorbate. Toxicity from heavy metals generates serious concerns in children and adults through ingestion, inhalation, and skin adsorption. The harmful health effects of heavy metals include neurological issues, musculoskeletal problems, and reproductive hormonal imbalances(Karić et al., 2022a).

Surface area analysis is crucial when studying biosorbents, which are materials, often of biological origin, used for the removal of pollutants from water or other solutions through a process known as biosorption.

CHAPTER 2

LITERATURE REVIEW

2.1 Napier Grass (Pennisetum Purpureum)

Napier grass, sometimes known as elephant or Uganda grass, is a major tropical feed crop. It is commonly employed in cut-and-carry feeding systems and is becoming increasingly important in other agricultural systems. It can withstand repeated cuts and swiftly rebound, producing excellent green shoots. It is a highly adaptable species that may be grown in a wide range of habitats and agricultural methods, including dry or wet climates, small-scale or industrial farming. It is a highly valued fodder, commonly employed in cut-and-carry systems across the tropics(Bajaj, n.d.).

Figure 2.1 : Napier grass manufactured
(Jamkhed et al., 2023)

2.1.1 Morphology of the Napier Grass

Napier grass is a tufted perennial grass with robust rhizomes. It has a robust root system that extends from the nodes of its creeping stolons. The culms are coarse, perennial plants that can reach heights of up to 7 metres and are branched above. Napier grass grows in bunches that can be up to one meter wide. The flat, linear, bluish-green leaves are up to 100-120 cm long, 1-5 cm wide, and hairy at the base. The leaf blade has a distinct midrib and a lovely serrated leaf edge. The inflorescence is a stiff terminal, bristly spike that can reach a length of 15-20 cm and is colored from yellow brown to purplish(Soetrisno et al., n.d.).

Spikelets arranged along a hairy axis fall as they mature. Due to its high productivity, Napier grass is an essential feed in the tropics. It's good for feeding cattle and buffalo. Napier grass is mostly used in cut-and-carry systems ("zero grazing") and is fed as stalls, silage, or hay. Napier grass can be grazed if it is kept in a rich vegetative stage: animals usually feeds just the younger leaves. Napier grass, as the name implies, is a vital source of food for elephants in Africa. So, because of that is normally known as elephant grass in Africa. (Mohammed et al., 2015).

2.1.2 Active compound of Napier grass stem

The developmental stages had notable impacts on the longest leaf length per plant and plant height, but did not influence the number of leaves per plant and the number of tillers. There was a significant increase in dry matter (DM) as maturity progressed, whereas crude protein (CP) and fat content showed a significant decrease. The acid detergent lignin (ADL), ash, acid detergent fibre (ADF), and neutral detergent fibre (NDF) exhibited no significant changes. As maturity advanced, there was a significant decrease in potassium, copper, and sodium, while calcium and zinc demonstrated a significant increase up to the intermediate stage before declining towards the late stage of maturity. The magnesium, phosphorus, manganese, and iron contents of the forage remained unaffected by maturity(Rambau et al., 2016).

The stem is underutilized because to its hardness, density, and high lignin concentration. The lignin composition of the Napier grass stem was recently established, providing insight into the crop's potential for chemical and biofuel generation. However, different regions of the plant have distinct cell types, tissues, and organs. As a result, these diverse sections of the plant exhibit varied reactions during pretreatment and have variable ideal pretreatment conditions, leading to different sensitivity to enzymatic hydrolysis(Chai et al., 2021).

Because of its high carbohydrate content of more than 60%, Napier grass stem has recently received a lot of attention as a possible lignocellulosic feedstock for biofuel generation (e.g., ethanol, butanol, and methane). However, lignocellulosic recalcitrance remains a significant barrier for the enzyme-based biorefinery business. The efficient pretreatment capable of deconstructing the recalcitrance of lignocellulosic material is considered a foundational step to achieve valorization based on the above-mentioned goal(Song et al., 2021).

2.2 Heavy metal toxicity

Heavy metals are a significant environmental hazard due to their mobility in aquatic habitats and toxicity to living organisms. These pollutants can be found in both surface and ground water. Even if they are present in minute, undetectable numbers, their recalcitrance and consequent persistence in water bodies suggest that concentrations may rise to the point where they exhibit dangerous qualities due to natural processes such as biomagnification(Riyazuddin et al., 2022).

These metals can either be found in various salt complexes or in their elemental condition, which indicates that they have not undergone any more biodegradative processes. Metal ions cannot mineralize in either circumstance. Metal recovery from industrial water must be considered technologically as well as environmentally. Heavy metals are considered part of the soil; yet, when too concentrated, they cause significant damage to the soil and plants. As a result, they are believed to be toxicants(Lichtfouse et al., 2005).

Heavy metal toxicity poses a significant threat, carrying various associated health risks. Despite lacking any biological role, these metals persist in forms that can be detrimental to human health and disrupt normal bodily functions. At times, they mimic essential elements in the body, acting as pseudo elements, while in other instances, they interfere with crucial metabolic processes. Certain metals, like aluminum, can be eliminated through natural processes, but others accumulate in the body and food chain, displaying chronic characteristics.

To address metal toxicity at different levels such as occupational exposure, accidents, and environmental factors various public health initiatives have been implemented for control, prevention, and treatment. The extent of metal toxicity is contingent upon factors such as the absorbed dose, the route and duration of exposure (acute or chronic), leading to the development of various disorders. Additionally, metal toxicity can result in substantial damage attributed to oxidative stress induced by the formation of free radicals. This comprehensive review provides insights into the mechanisms of toxicity for specific heavy metals and their associated health effects. (Monisha Jaishankar. et. al.)

Table 2.1: Type of heavy metals and health effects on human

(Source: Narjala R. J., 2020)

Heavy metals	Source	Health effect
Arsenic	Atmospheric deposition Mining pesticides	Degenerative, inflammatory and neoplastic changes of skin, respiratory system, blood, lymphatic system, nervous system and reproductive system.
Lead	Gasoline, house paint, plumbing pipes, pewter pitchers, storage batteries, toys and faucets.	Serious effect on mental health (Alzheimers disesase), Nervous system
Mercury	Coal combustion, Fish, Mining, Paint industry, Paper industry, Volcanic eruption	Sclerosis, Blindness, Minamata disease, Deafness, Gastric problems, Renal disorder
Cadmium	Plastic, Fertilizers, pesticides	Osteo related problems , Prostate cancer, Lung diseases, Renal issues
Chromium	Steel fabrication, Electroplating, Textile	Lung disorders (bronchitis,cancer), Renal and reproductive system
Zinc	Oil Refining, Plumbing, Brass manufacturing	Gastrointestinal disorders, Kidney & Liver abnormal functioning
Iron	High intake of iron supplements & oral consumption	Vomiting, Diarrhea, Abdominal pain Dehydration & lethargy
Copper	Copper polishing, Plating, Printing	Abdominal disorders, Metabolic activity abnormalities

2.3 Adsorption analysis

Adsorption mechanisms are determined by the physicochemical parameters of the adsorbent, heavy metals, and working circumstances such as, temperature, adsorbent amount, pH value, adsorption time, and initial concentration of metal ions. Generally, heavy metal ions can be adsorbed onto the adsorbent surface, as illustrated in figure 2.2. Adsorption defined as the interaction of a fluid (adsorbate) and a solid surface (adsorbent), takes place on the surface of a solid due to the attraction of the atoms or molecules to the surface of the solid (Qasem et al., 2021b).

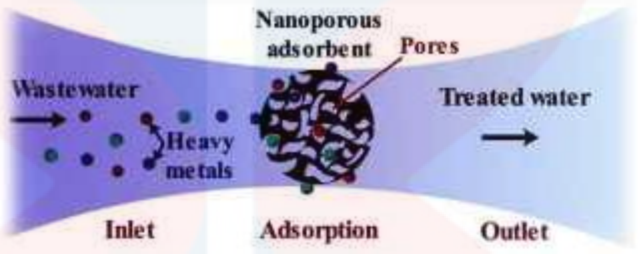


Figure 2.2: Heavy metal ion adsorption method

Natural materials, including waste from industrial and agricultural activities, can be used as low-cost adsorbents to remove heavy metals. Cost is a significant consideration when evaluating different adsorbent materials. Adsorbents might be considered 'low-cost adsorbents' if they require minimal processing, are readily available, or are waste materials from other industries or agriculture(Chakraborty et al., 2022).

Biomaterials are cost-efficient and easy to prepare, making them excellent at removing heavy metals from wastewater. Modifying biomaterials with physical and chemical activation can improve their surface area and porous structure. More in-situ trials are needed to assess its effectiveness and environmental impact before widespread use. Adsorption is a potential approach for removing heavy metals from groundwater and industrial waste because to its economic feasibility, low power consumption, and minimal formation of chemical byproducts and sludge (Gupta et al., 2021).

Percentage removal efficiency was calculated using the following equations respectively:

$$\text{Removal efficiency (\%)} = \frac{C_0 - C_c}{C_0} \times 100 \qquad \text{Equation 2.3(a)}$$

Where, C_0 is the initial of concentration metal ion in solution; C_c is the metal concentration in solution at equilibrium. The removal efficiency is usually expressed as a percentage, providing insight into what proportion of the initial concentration has been removed or reduced by the treatment or removal process. A higher removal efficiency indicates a more effective removal process.

2.4 Importance of Pretreatment

Some pretreatment techniques may improve the adsorption capacity of biomass materials. Autoclaving is another technique of pretreatment that can degrade fungal structure and disclose the possible colour of binding sites. Scanning Electron Microscopy was the preferred analytical tool for optimizing numerous parameters and expecting greater performance prior to and after adsorption. The approach determines optimal settings under predefined factor preferences, such as high removal at the ideal condition or the least number of experiments(Wahab RA et al., 2014).

Pretreatment were primarily done to increase efficiency and adsorption rate, and it can be done in a variety of ways. Desorbing eluents can be used to recover adsorbed pollutants, and biosorbent regeneration can be done chemically, thermally, or electrochemically(P.R. Yaashika et al.,2021). Advances in biosorbents for pollution removal. Pretreatment is required to improve the materials' ability and usability. A single biological adsorbent material is insufficient to meet the requirements of current applications. Several biosorbent pretreatment techniques have recently appeared. Biosorbents' sorption performance can be increased by extending the adsorption sites on their surface and maximising the functional groups present on the surface of the adsorbent material(Benis et al., 2020). Physical pretreatment commonly includes fragmentation and heat, whereas chemical pretreatment includes acid-base treatment, oxidant, inorganic salts, and organic solvents treatments.

2.4.1 Methods of biomass pretreatment

Each pretreatment process is designed to achieve a specific effect, such as improving chemical surface heterogeneity, increasing the number and dispersion of functional groups available for binding with the metal, and altering the surface morphology. Therefore, the selection of a suitable pretreatment method should be based on the specific metal ion targeted for removal. The most valuable and extensively researched method of pretreating leaf biomass is chemical modification, primarily due to its low cost and procedural simplicity. This process is typically a one-step procedure in many cases.

Here's a general overview of methods used in leaf biomass pretreatment such as mechanical methods which include milling or grinding. In general, the step by physical reduction of leaf biomass into smaller particles increases the surface area, facilitating further chemical treatment. Other than, milling with ball or Roller Mills can impact and compression forces break down the structure, reducing particle size. Pretreatment undergo by the chemical methods somehow using the acid pretreatment which is the treatment with dilute acids, such as sulfuric acid or hydrochloric acid, can hydrolyze hemicellulose and disrupt the lignin structure.

Furthermore, alkaline pretreatment means the use of alkalis like sodium hydroxide or ammonia helps break down lignin and improves enzymatic digestibility. Originally, pretreatment one of the method using organic solvents, such as ethanol or methanol, are used to dissolve or disrupt the lignin, making cellulose more accessible. Oxidative pretreatment such a treatment with peroxides or ozone can break down lignin and hemicellulose, improving enzymatic hydrolysis efficiency.

Many more and easier the research found is the pretreatment by the physical methods. One of the method is steam explosion. It described about the biomass is exposed to high pressure steam and then rapidly depressurized, causing the fiber to swell and disrupt, enhancing accessibility to enzymes. Second include microwave and radiofrequency pretreatment using the electromagnetic waves generate heat, causing internal changes in the biomass structure, making it more amenable to subsequent processes. Ultrasound pretreatment also example of the pretreatment for the biomass. It done by ultrasonic waves create cavitation, leading to the physical breakdown of biomass components(Ahmed et al., 2023).

2.5 Operational parameter

2.5.1 Effect of initial metal ion concentration

The initial concentration of metal ions in solution determines the amount of metal ions adsorbed by the sorbent when binding sites are accessible. The increase in absorption capacity with increasing initial metal concentration is attributed to increased metal ion availability for sorption. Increased concentration produces more collisions between metal ions and sorbent, which is a significant element in kinetics for boosting the velocity of chemical processes(Vimala and Das, 2009). The initial rapid increase in efficiency is due to the availability of binding sites on the biomass, which decreases as metal ion concentration increases. As a result, the sorption reached saturation as the set number of binding sites was depleted.

2.5.2 Effect of adsorbent dose

Different biosorbent doses had minimal effect on the results; for example, 5 g of biosorbent showed higher uptake. As a result, it is expected, as increasing adsorbent doses increases surface area. While the use of 5 g/L adsorbent dose is economically viable. This trend is expected because as the adsorbent dose grows, so does the number of adsorbent particles and hence the amount of metal ions attached to their surfaces. Although the initial metal ion concentration remains constant, increasing the adsorbent dose might increased the surface area for sorption, while decreased metal ion adsorption per unit mass of adsorbent. Adsorbent dose is an important quantity because it affects an adsorbent's capacity at a particular initial concentration of the adsorbate under working conditions. As adsorbent dosage increases, more surface area becomes accessible for adsorption. This increases the number of active sites on the adsorbent, allowing metal ions to penetrate more easily(Azouaou et al., 2010).

2.5.3 Agricultural waste heavy metal adsorption

Since the 1960s, agricultural production has tripled due to increasing land utilization and technical breakthroughs from the green revolution, resulting in higher yields to satisfy the demands of a growing global population(Karić et al., 2022b). Agricultural waste typically contains cellulose, hemicellulose, and lignin as main polymers. Using agro-wastes is a sustainable and eco-friendly way to effectively use plant waste(Kumar et al., 2023). In general, agricultural wastes are classified as biomass residues and can be separated into two types: crop residues and agro-industrial residues(Collivignarelli et al., 2022).

Table 2.2: Biosorbent derived by agricultural waste removal efficiency based on operational parameter

Adsorbent	Heavy metal	Removal range parameter	References
Pennisetum purpureum	Cadmium	pH: Increase pH 1 to pH 4 Initial conc.: 5 mg/L to 92.3% Contact time: 30 minutes	Shing et al., 2019b
Rice husk	Arsenic	pH: 7 Temp : 20 °C	Collivignarelli et al., 2022
Orange peels	Cadmium	pH :5.5 Temp: 45 °C Dosage: 0.06 g	Akinhanmi et al., 2020
Neem leaves	Copper	pH: 7 Dosage: 1.0 g Initial conc.: 110 ppm	Al Moharbi et al., 2020)

2.6 Importance of surface area analysis

The importance of surface area analysis in biosorbents lies in several key aspects include adsorption capacity where is the surface area of a biosorbent directly affects its adsorption capacity. A higher surface area provides more sites for adsorption, allowing the biosorbent to capture and hold a greater amount of target substances, such as heavy metals, dyes, or other pollutants(Obi et al., 2023). Other than influenced by interaction sites which is the surface area determines the number of active sites available for interaction with pollutants. In biosorption, these active sites are typically functional groups on the biosorbent's surface.

Analyzing surface area helps understand the distribution and availability of these sites for effective adsorption(Allou et al., 2023). Furthermore, efficiency and effectiveness is closely linked to surface area. A biosorbent with a larger surface area can interact with a higher volume of contaminants, making the process more effective in treating polluted water or wastewater(Dey et al., 2021). Surface area analysis is essential for understanding the kinetics and rate of adsorption. A biosorbent with a well-characterized surface area allows researchers to study how quickly pollutants are taken up by the biosorbent, providing insights into the effectiveness of the process.

Knowledge of surface area is crucial when optimizing biosorption processes. Surface area analysis facilitates the comparison of different biosorbents. Researchers can evaluate and select biosorbents based on their surface characteristics, helping to identify the most suitable materials for specific applications. Moreover, surface area analysis is an integral part of biosorbent characterization. It provides valuable information about the physical structure and properties of the material, aiding in understanding its behavior during biosorption. Overall, surface area analysis is essential for optimizing biosorption processes, improving the efficiency of pollutant removal, and advancing the field of sustainable water treatment.

Table 2.3 shows the differences of surface area analysis from various agricultural waste for heavy metal removal.

Table 2.3 : Surface area analysis various agricultural plant

Biosorbent	Surface area (m2 /g)	Removal element	References
Modified Napier grass	271.13	Crude Oil	(Obi et al., 2023)
Unmodified Napier grass	180.07	Crude Oil	(Obi et al., 2023)
Raw corn cob	5.87	Cr(VI)	(Allou et al., 2023)
Activated Corn Cob	14.85	Cr(VI)	(Allou et al., 2023)

Fresh Orange peels	45.42	Nitrate	(Dey et al., 2021)
Aloe Vera	13.8	Zn (II)	(Moosa et al., 2016)

Surface area analysis is the measuring of a particle's accessible surface. It is significant because it is the mechanism via which a solid interacts with its surroundings, whether they are gases, liquids, or other solids. Surface area generally increases when particle size reduces; but, if porosity is formed, the increase in surface area can be far greater than that provided by size reduction. Acetylation increases surface area and pore volume by replacing low-weight hydroxyl groups with bulkier acetyl groups from acetic anhydride, leading to increased active area(Obi et al., 2023).

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Napier Grass was used as an absorbent directly without any prior treatment to remove the heavy metal in the industrial wastewater. The sample water was prepared (Kawasan Perindustrian Jeli). The present of heavy metal in wastewater were determined by Atomic Absorption Spectrophotometer

3.2 Apparatus and Equipment

The apparatus and equipment used for Napier grass as biosorbent to heavy metal removal are summarized in Table 2.4 and Table 2.5.

Table 2.4: List of apparatus used in research

No.	Apparatus
1.	Mortar and pestle
2.	Beaker 50 mL
3.	Sieve 125 µm
4.	Schott bottle 1 L
5.	Measuring cylinder 10 mL
6.	Volumetric flask 250 mL
7.	Falcon tube
8.	Filter funnel

Table 2.5: List of equipment used in this research

Equipment	Purpose
Oven drying	Remove moisture of plant material
Grinder	Grinding up plant sample into a smaller substance
Incubator shaker	Incubate and shake samples to properly metal ion adsorbed on the biosorbent
Atomic Absorption Spectrometer	To analyze the presence of heavy metal ion
Surface Area Analyzer	To analyze the specific surface area and pore size of the biosorbent

3.3 Methods

3.3.1 Pretreatment of plant sample

The Napier Grass was used for the experiments and was collected from the Kampung Baru, Jeli. Napier grass were used as a natural material. The matured and the width one was been selected. After an initial drying stage at ambient temperature, it was been roughly grinded with a mortar and pestle. Then important part grinder was used to make sure the Napier grass properly treated in powder form in order to increase the specific surface of Napier grass. Clean Napier grass was extensively dried to constant weight in an oven at approximately 60°C for approximately 24 hours. After that, the particle size 125 µm was sieved for further experiment. Later on, this adsorbent was oven-dried at 65°C for 4 h.

3.3.2 Wastewater samples

Industrial wastewater samples were collected from sawmill manufacturing factories in Jeli. The samples were collected into 1 L Schott bottles already rinsed with distilled water. They were tightly sealed and kept in the refrigerator before use. Heavy metal identified using a Perkin Elmer Atomic Absorption Spectrophotometer, the absorbance of the wastewater solutions were measured alongside that of the standard solutions. The metal concentrations (in mg/L) were calculated directly from a calibration curve generated after blank correction. The same approach was used to analyse reference heavy metal standard solutions (Johnson Mattley Material Technology) to ensure the quality of the results. 50 mL water sample was added into beaker. Final process diluted sample run the AAS analysis. The heavy metal in diluted water sample were calculated.

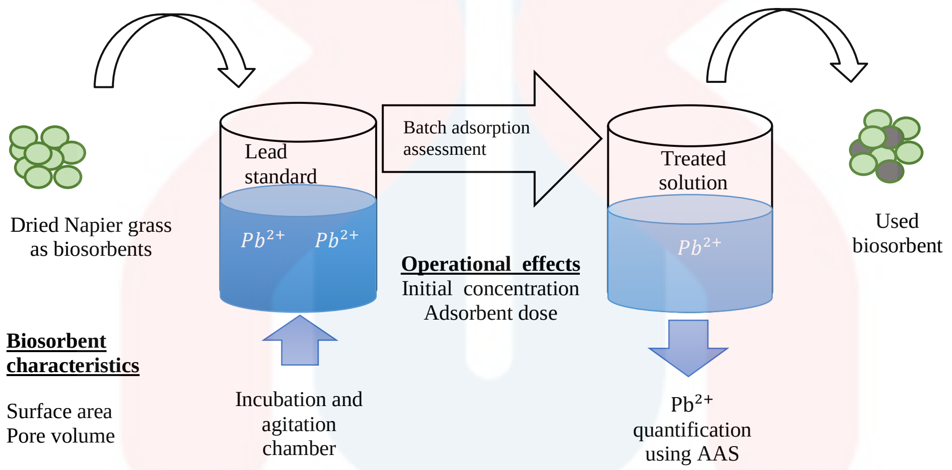


Figure 2.3: The schematic batch mode experiment to assess the efficiency of biosorbent Napier grass for Pb2+ ion sorption.

3.3.3 Preparation of initial metal ion concentration

An equilibrium experiments were been carried out the heavy metal scavenging ability of the Napier grass biosorbent. For the equilibrium experiments, 500 miligram of the lead nitrate Pb(NO3)2 was weighed then transferred to the 500 mL volumetric flask and distilled water has been added until the equilibrium layer to make 0.5 mg/L lead stock solution. The final concentration of heavy metals in the filtrate was determined by AAS. (Asibiojo O.I. et al., 2010). The process was repeated since prepared the 0.6 mg/L and

0.7 mg/L lead stock solution. Adsorption tests were conducted at 25°C at natural pH of solution and adsorbent dose with 2 gram.

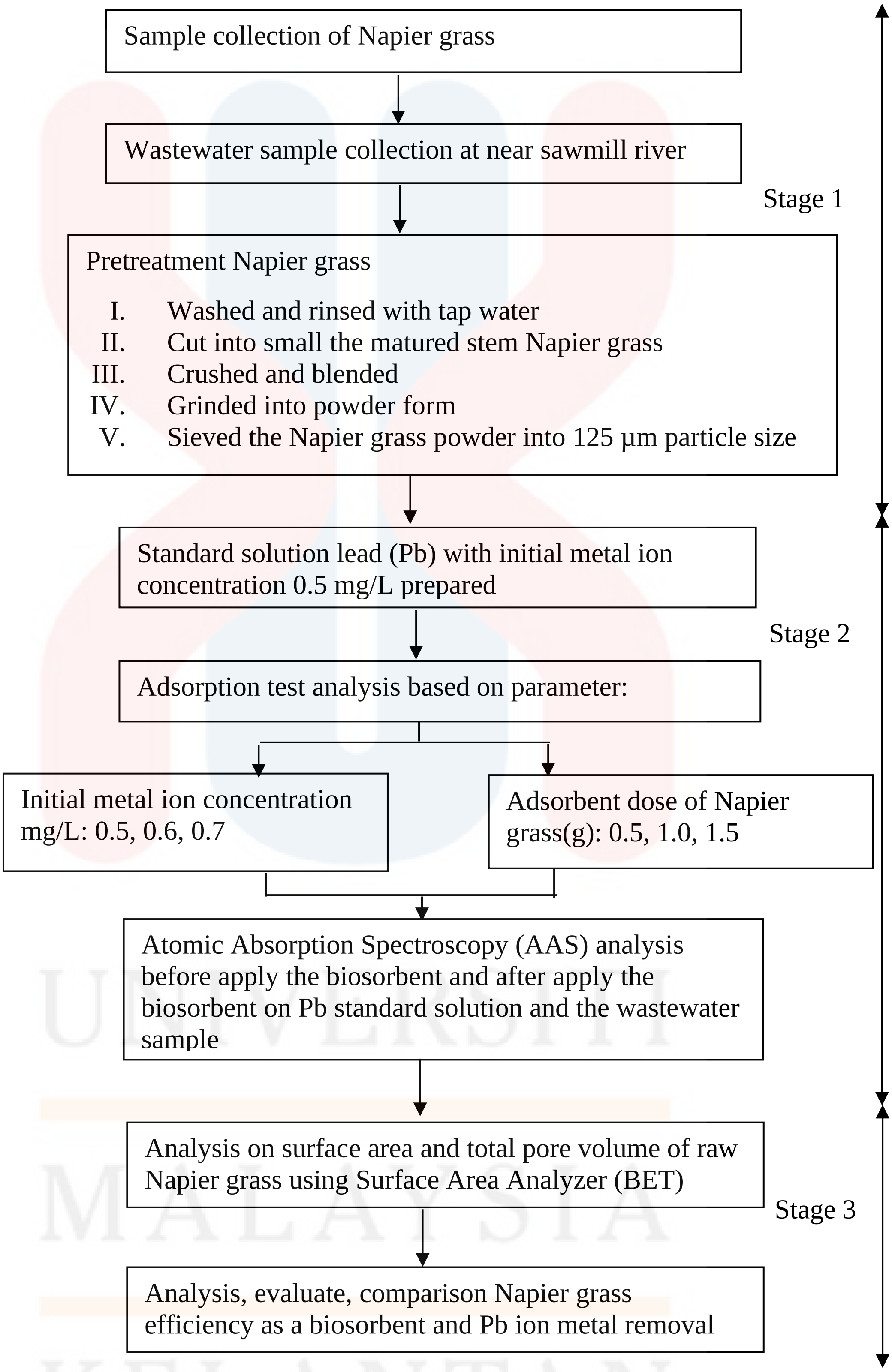
3.3.4 Preparation of adsorbent dose

The influence of adsorbent dosage on the percentage of metal removed from water samples. Adsorbent dosage is also a significant component in determining adsorbent's ability to absorb heavy metals. Increasing the dose of adsorbents typically increases adsorbed capacity until a limit is reached. If the dose is increased further, the adsorption capacity will be constant. The effect of adsorbent dosage on metal adsorption was investigated since it impacts the capacity of the adsorbent for a particular initial metal concentration(Shing et al., 2019).

The biosorption of lead ions was investigated using the batch method to determine the effect of initial metal ion concentration and adsorbent dosage. For the fixed variables at the start of the experiment, 0.5g Napier grass was mixed with 50mL of 0.5 mg/L lead solution in a 250 mL Erlenmeyer flask and aggressively agitated with an incubator shaker. The shake speed was held constant at 100 rpm for 10 minutes. The variables could be altered depending on the best condition attained.

This study also looked at adsorbent dosage (1.0 g and 1.5 g) affected the results. Samples were taken from the flasks and the residual lead ion concentration in each flask was determined. The suspensions were filtered through Whatman filter paper, and the supernatants were examined using Atomic Absorption Spectroscopy to determine absorbance at the maximum wavelength of 217nm after one week. The percentages of removal of lead ions were calculated using Equation 2.3.

3.4 Research Flowchart



CHAPTER 4

RESULTS AND DISCUSSION

The parameter adsorbent dosage and initial concentration heavy metal ion on the removal heavy metal using Napier grass, atomic absorption spectroscopy for the analysis the presence of heavy metal in wastewater, Surface Area Analyzer on the Napier grass as an biosorbent and the raw Napier grass which effective in adsorption of the heavy metal.

4.1 The Influence of Initial Metal Ion Concentration

The amount of metal ions biosorbed by the sorbent depends on their initial concentration in solution and accessible binding sites. This is evident in the results shown in Figure 2.4. Based on the result obtained, the removal rate increased from 77.04% , 88.18% and 89.97% at 0.7 mg/L , 0.6 mg/L and 0.5 mg/L respectively.

The initial concentration plays a crucial role in the efficiency and effectiveness of the adsorption process. The analysis results indicate that the removal rate of Pb or other heavy metals diminishes as their concentration increases. At lower concentrations, there is a significant ratio between the adsorption sites of the adsorbent and the molecules of heavy metals. Consequently, the fractional adsorption becomes independent of the initial concentration. However, at higher concentrations, the number of available adsorption sites decreases. As a result, the percentage adsorption of heavy metals like Pb, which is contingent upon the initial concentration, decreases. This could be attributed to the elevated initial ion concentration, which creates a more substantial driving force capable of overcoming all mass transfer resistances between the solid and aqueous phases. As a consequence, there is a higher adsorption of copper ions during the initial stages.

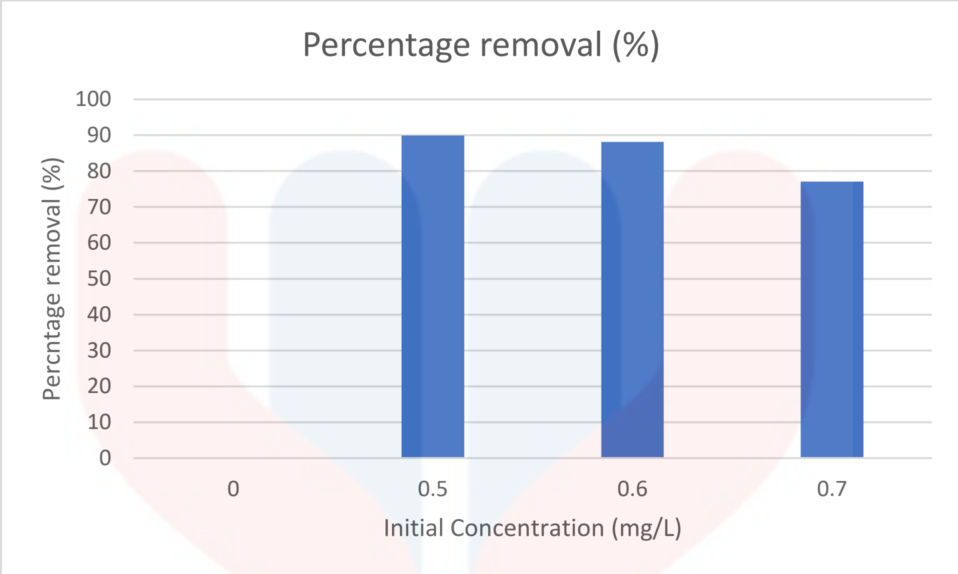


Figure 2.4 : Effect of initial metal ion concentration on the adsorption capacity of Pb

4.2 The Influence of Adsorbent Dose

Based on Figure 2.5, the adsorbent dose of 1.0g and 1.5g has the highest removal efficiency of 79.62%. It was also obtained that at conditions of pH natural and an initial concentration of 0.5 mg/L. Adsorbent dose is a significant factor affecting the adsorption capacity. The more adsorbent added, the higher the efficiency of % removal because of the surface area of the adsorbent(Malacas et al., 2019). The large amounts of active sites on the adsorbent surface had led to the more metal ion adsorption rate initially, which was typically influenced by diffusion from the bulk solution to the adsorbent surface.

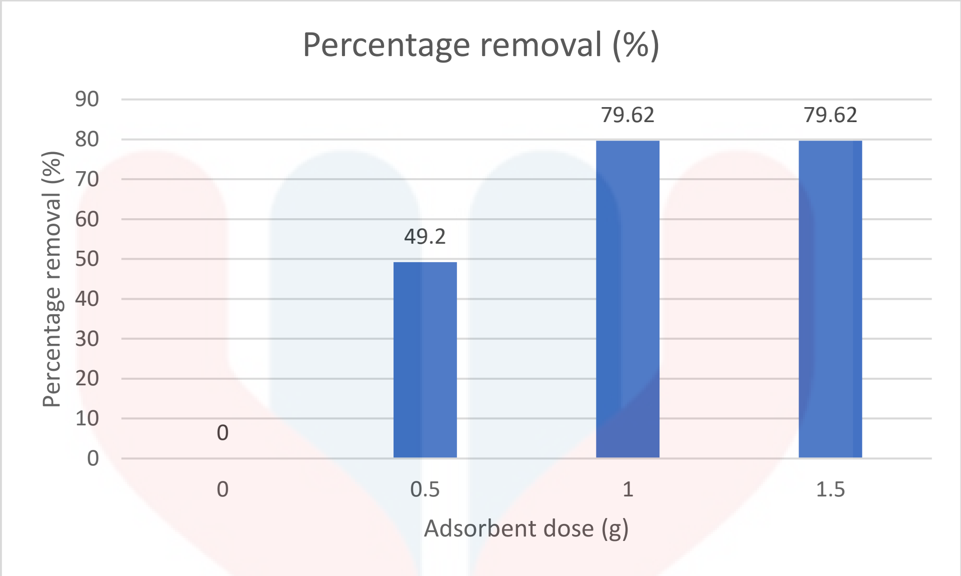


Figure 2.5: Effect of adsorbent dose on the adsorption capacity of Pb

4.3 Atomic Absorption Spectroscopy Analysis

Table 2.6 : Effect of Napier grass dosage on removal of heavy metals at 0.5 mg/L of Pb

Adsorbent Dose	Absorbance	Concentration
0.0g	0.1038	2.073
0.5g	0.0127	0.254
1.0g	0.0037	0.074
1.5g	0.0037	0.047

The primary reason for this is that when the adsorbent mass grows, more adsorption sites are available per mass of adsorbent surface, increasing the total quantity of metal removed. This finding suggests that Pb ion removal occurs primarily in the early stages, and that the functional group in the Napier grass biosorbent can influence heavy metal adsorption capability by determining the availability of adsorption sites.

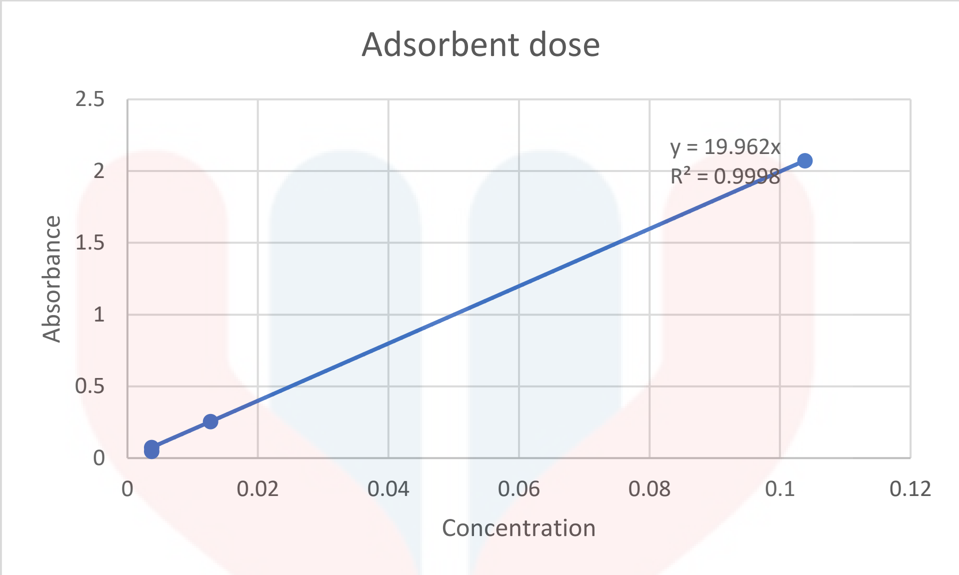


Figure 2.6: Effect of dosage of Napier grass on removal of Pb ion.

The dosage of biosorbents has a significant impact on Pb(II) sorption at a specific starting concentration. Sorption is connected to mass transfer. As the contact between the sorbent and the sorbate increases, so does the sorption capability. However, excessive use of sorbent raises the cost of the sorption process, making contact less appealing(Heraldy et al., 2018).

Figure 2.6 shows the findings of investigating the dosage dependency of biosorbents on Pb(II) sorption. Figure 2.6 shows that the sorption of Pb(II) increased with sorbent dosage from 0.5 g to 0.15 g/50 mL, reaching a maximum of 79.62% elimination for NG biosorbent. Subsequently, as the sorbent dosage was increased, the amount of Pb(II) removal was shown to decline due to the. NG dosage has a significant impact on the sorption of Pb(II). Subsequently, when sorbent dose increased, the amount of Pb(II) removal decreased due to the overlaying or aggregation of active sites. The rise in NG dosage corresponds to the accessibility of various active sites for the joining of Pb(II) in solution, which supports the relationship between removal rate and NG dosage.

Table 2.7 : Effect of initial ion concentration Pb solution at 2gram of Napier grass

Initial Concentration	**Concentration**	**Absorbance**
0.5 mg/L (Before apply biosorbent)	2.073	0.1038
0.5 mg/L (After apply biosorbent)	0.476	0.1013
0.6 mg/L	0.245	0.0123
0.7 mg/L	0.208	0.0104

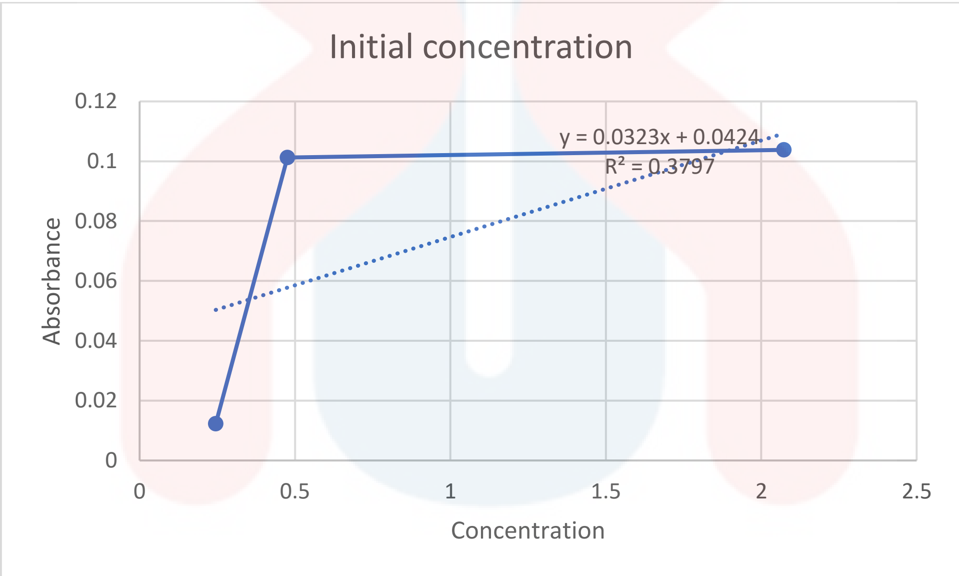


Figure 2.7 : Effect of heavy metal removal on initial metal ion concentration Pb solution

As the metal ion concentration increases, the removal capability decreases due to binding site saturation. This is owing to increased initial ion concentration, which provides a stronger driving force to overcome all mass transfer resistance between the solid and aqueous phases, resulting in enhanced metal ion adsorption.

In the context of heavy metal removal, absorbance measurements are often used to quantify the concentration of heavy metals in a solution. By establishing a calibration curve based on known concentrations of heavy metals, one can then use absorbance measurements to estimate the concentration of heavy metals in an unknown sample. So the Figure 2.7 showed that the absorbance and the concentration not linear and the deviation posses 0.4822. It might occurred in solution condition without digestion. Either it contains interfering substances during the filtered nor the high concentration during the serial dilutions. In such cases, adjustments or corrections may be needed for accurate quantification especially during the adsorption process so that the removal of heavy metal efficiently continued.

4.4 Presence copper and lead in wastewater

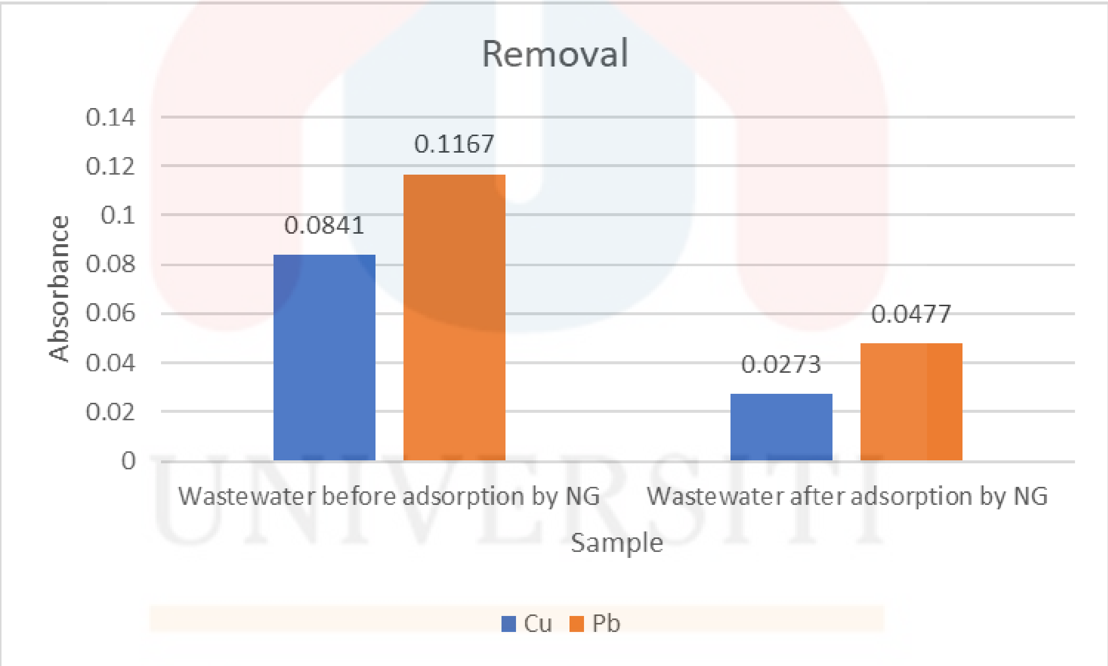


Figure 2.8: Different the percentage of copper and lead in the wastewater before and after apply the NG.

Table 2.8: Percentage removal Cu and Pb in the wastewater after treated with NG

Sample	Percentage Removal (%)
Cu	58.29
Pb	59.14

Cu is often found in high amounts in wastewater because it is widely used in industrial applications such as metal polishing, electroplating, polymers, and etching. Furthermore, even at low quantities, copper is a highly toxic metal, and copper-contaminated wastewater must be cleaned before it is released into the environment(Ab Hamid et al., 2022). Copper concentration in the wastewater sample is higher, suggesting that copper contamination is from anthropogenic sources.

Timber treatment chemicals found elevated concentrations of copper(Ugbune, 2020). Heavy metals are recognized carcinogens, and wood dust has been linked to carcinogenic consequences because dust formed by woodworking often contains a high proportion of particles that are deposited in the nasal cavity, making this study necessary. The goal of this study is to quantify heavy metal concentrations in wastewater from sawmills in order to provide information about the impact on human health and the environment.

Table 2.7 stated percentage removal of copper in wastewater using the raw Napier grass showed 58.29% while percentage removal of lead in wastewater after apply the raw biosorbent is 59.14%. The concentrations of Pb in wastewater higher than Cu but both of the heavy metal ion showed the removal efficiency mostly adsorbed by the raw NG with 2 gram.

4.5 Surface Area Analyzer

The sorption capacity of a material is also determined by its chemical structure, porosity, specific surface area and diffusion characteristics. Furthermore, the adsorption process is influenced not only by adsorbent qualities, but also by shape, chemical composition, water solubility, and pollutant polarity. Last but not least, adsorbents must have various functional groups that can bind heavy metal ions from wastewater during the removal process(Ab Hamid et al., 2022). The specific surface area, pore diameter average and pore volume of rNG determined BET method were presented in Table 2.9.

Table 2.9: Surface area analysis raw NG

Total pore volume (P/Po)	Surface area (m^2/g)	Average pore Diameter (Å)
0.994	2.360	9.590

The given application or study may have unique criteria or expectations for the adsorbent's surface area. Values below standard may be important for the intended purpose, depending on the properties of the pollutants being targeted or the nature of the adsorption process. The surface area of a NG directly affects its adsorption capacity. A lower surface area provides less sites for adsorption, allowing the NG to capture and hold a smaller amount of lead and copper.

The values mentioned could be part of an experimental parameter set to observe how surface area affects the adsorption efficiency. Researchers might be exploring a range of surface area values to understand the optimal conditions for raw Napier grass powder as an adsorbent without any modification. The pore sizes are divided into three categories according to the IUPAC criteria: micropores (less than 20 Å or 2 nm), mesopores (between 20 Å or 2 nm and 500 Å or 50 nm), and macropores (greater than 500 Å or 50 nm). This shows that the raw NG were dominated by mesopores. The slight reduction in pore size was likely due to the swelling of the cell wall of the Napier grass during the acetylation process through the introduction of the bulky acetyl groups(Obi et al., 2023).

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In conclusion, the imperative need to address the adverse impacts of heavy metals on human health and environmental organisms underscores the significance of eliminating lead and copper from industrial effluents, making it a pivotal focus in contemporary environmental research. Over the past few years, diverse treatment approaches, encompassing physical, chemical, and biological methods, have been explored, with a primary emphasis on adsorption. However, it is crucial to exercise caution, considering factors like the initial concentration of lead and copper ions, operational costs, and the environmental repercussions and compatibility of the implemented methods. Further research is warranted to enhance specific aspects of the current techniques. Notably, the extended duration required by existing cementation agents to remove higher concentrations of lead and copper ions from wastewater necessitates exploration into new agents that can expedite the process. Recent studies have indicated the potential efficacy of biosorption and natural adsorbents in treating lead and copper pollutants. Hence, evaluating novel forms of biosorbents is essential to maximize effectiveness, given their advantages such as low raw material costs, strong adsorption performance, and eco-friendliness. This approach holds promise for the removal of lead and copper. To advance the understanding of copper treatment in wastewater, it is imperative to address existing knowledge gaps through further investigation. The objective of this study was achieved successfully. Firstly, effect of initial concentration heavy metal ion for heavy metal removal using raw NG showed the maximum removal efficiency percentage 89.97%. In determination of initial heavy metal ion concentration, the best conditions were 0.5 mg/L which the smallest. Besides, the second objective, the effect of the adsorbent dosage for heavy metal removal was evaluated with maximum removal efficiency 76.62%. Adsorbent dosage NG for heavy metal removal gave the increasing value respectively because of the surface area and pore size.

5.2 Recommendations-

The following suggestions for future work are recommended for future development:

1. Cellulose can be modified in two ways: directly chemically or by grafting monomer to the cellulose backbone. Chitin and chitosan could be physically or chemically modified to improve their heavy metal adsorption ability and robustness under extreme pH conditions.
2. pH seems to be the most important parameter in the biosorptive process: it affects the solution chemistry of the heavy metals, the activity of the functional groups in the biomass and the competition of heavy metal ions.
3. The most efficient method of digestion applicable to all the heavy metals in the study was therefore Nitric Acid at 100 °C for 120 minutes for Lead and Copper.

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APPENDIX A

Figure A.1: The dried sample before used.

Figure A.2: The dried sample after used.