Removal of Nickel From Aqueous Solution Using Rambai Stem

by

Noor Khuzaimah Binti Ahmad

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CERTIFICATION OF APPROVAL

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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NOOR KHUZAIMAH BINTI AHMAD

ABSTRACT

Heavy metals contamination in water and soil had become an issue ever since the start of the industrialization in many countries around the world. The issue continuously attracts our concern as it brings so many health effects and furthermore, altering our ecology. Thus, heavy metals treatment becomes inevitable. Many types of treatment had been introduced in hope of reducing such contamination in our environment. Biosorption using biomass and other biological sources is one of the promising methods being develop.

An effort has been made in this study to evaluate the potential of Rambai stem in removing Nickel, as the heavy metal of concern in waste water, as well as to provide a view on the conditions that can permit a higher removal percentage. The removal effect was found to be higher at pH range of 4 to 5, adsorbent dosage of 0.2g and initial concentration of heavy metal solution of 10 ppm. From the experimental results evaluation, it can be concluded that the removal of Nickel was higher at a weak range of acidic solution, a higher dosage of adsorbent and at the lowest initial concentration of heavy metal solution. The adsorption data obtained during the study fitted well in Freundlich Isotherm, thus signifies that the adsorption of Nickel using Rambai stem follows a heterogeneous surface adsorption.

The project can bring a huge advantage to the community since the project is involving Rambai plant which can be found almost abundantly in our country. The preparation of Rambai stem to be used as the adsorbent is quite simple, yet the results obtained from the experiment can be develop further and become a reference for the future research in the biosorption involving other type of plant's stem.

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ABBREVIATIONS AND NOMENCLATURES

Ni	= Nickel
ppm	= Part per million (concentration = mg/L)
V	= Volume of metal-bearing solution contacted with the sorbent. [L]
C_I	= Initial concentration of the metal in the solution [mg/L]
C_f	= Equilibrium concentration of the metal in the solution [mg/L]
S	= Amount of added sorbent on dry basis. [g]

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

1.1.1 Heavy Metals in Environment

Generally, metals are classified as heavy metals when their associated densities are higher than 5 gcm^3. Heavy metals are major pollutants in marine, ground, industrial and even treated wastewaters. The release of large quantity of heavy metals into natural environment had resulted in serious environmental problems and at the same time, posed significant impacts to human health. This is due to the non-biodegradability and persistence of heavy metals, which can interfere with the environmental elements such as the living organisms' food chain.

Over the centuries, the heavy metals contamination in wastewater had become one of the largest contributors to soil and water pollution. Main sources of heavy metal contamination include urban industrial aerosols, solid waste from animals, mining activities, industrial and agricultural chemicals.

Heavy metals, even at low concentration can posed a serious threat to human health. This is due to the non-biodegradability of heavy metals and their ability to leach out from waste dumps, polluted soils and waters into human and other living organism's food chain. Thus, their treatment becomes inevitable.

Many techniques exist in treating heavy metals in contaminated waters such as the reverse osmosis, electrodialysis, ultrafiltration, ion exchange, chemical precipitation and many others.

Reverse Osmosis: It is a process in which heavy metals are separated by a semi-permeable membrane at a pressure greater than osmotic pressure caused by the dissolved solids in wastewater. The disadvantage of this method is that it is expensive.

Electrodialysis: In this process, the ionic components (heavy metals) are separated through the use of semi-permeable ion selective membranes. Application of an electrical potential between the two electrodes causes a migration of cations and anions towards respective electrodes. Because of the alternate spacing of cation and anion permeable membranes, cells of concentrated and dilute salts are formed. The disadvantage is the formation of metal hydroxides, which clog the membrane.

Ultrafiltration: They are pressure driven membrane operations that use porous membranes for the removal of heavy metals. The main disadvantage of this process is the generation of sludge.

Ion-exchange: In this process, metal ions from dilute solutions are exchanged with ions held by electrostatic forces on the exchange resin. The disadvantages include: high cost and partial removal of certain ions.

Chemical Precipitation: Precipitation of metals is achieved by the addition of coagulants such as alum, lime, iron salts and other organic polymers. The large amount of sludge containing toxic compounds produced during the process is the main disadvantage.

Phytoremediation: Phytoremediation is the use of certain plants to clean up soil, sediment, and water contaminated with metals. The disadvantages include that it takes a long time for removal of metals and the regeneration of the plant for further biosorption is difficult.

These methods have associated with disadvantages such as incomplete metal removal, high reagent requirement, generation of toxic sludge or other waste products that require careful disposal (Ahalya et al., 2003) and due to the legal constraint and increasing environmental awareness, a need for cost-effective alternative technologies is essential. In this endeavor, biosorption has emerged as an option for eco-friendly and cost-effective method to accomplish the purpose. Biosorption had seemed to be a promising alternative for treating heavy metals contaminated waters, more efficiently in treating waters associated with trace concentration of heavy metals. This technique employs various types of biomass or other agricultural sources to trap heavy metals in contaminated waters.

1.1.2 Biosorption

Biosorption is a biological process of removing heavy metals which involves a solid phase (sorbent; usually biological material) and a liquid phase (solvent; normally water) containing a dissolved species to be adsorbed (adsorbate, a metal ion). Due to higher affinity of the sorbent for the adsorbate species the latter is attracted and bound with different mechanisms. The process continues until equilibrium is established between the amount of solid-bound adsorbate species and its portion remaining in the solution.

In the past few years a lot of efforts have been made on screening of efficient biomass types, its preparation and biosorption mechanism determination. The advantage offered by biosorption method among others is the effectiveness in treating a large volume of effluents with low concentration of pollutants. Other than that, the method also offers some other advantages such as cost effectiveness, minimization of chemical/ biological sludge, requirement of additional nutrients and regeneration of biosorbent with possibility of metal recovery.

Sources of Biomass for Biosorption

- Seaweeds
- Microorganisms (bacteria, fungi, yeast, molds)
- Activated sludge
- Fermentation waste
- Other specially propagated biomasses.

1.3 Adsorption Isotherms

ıe important aspect in evaluation of adsorption is the equilibria of the adsorption. Equilibrium ıdy will give the capacity of the adsorbent. The adsorption isotherms (usually the ratio between the quantity adsorbed and that remaining in solution at a fixed temperature at equilibrium) are used to describe the equilibrium relationship. Most frequent biosorption equilibria are described with adsorption isotherms of Langmuir or Freundlich types

1.1.4 The Adsorbent – Rambai stem (*Baccaurea motleyana*)

Rambai or scientifically referred as *Baccaurea motleyana* is a type of fruit that is believed to be originated from South East Asia. It is widely cultivated throughout Peninsular Malaysia, Sumatra, Borneo, Java and Bali, and has found its way to neighboring countries like Thailand and the Philippines.

The plant is normally cultivated for the fruit with so little prospect for other uses. The wood is of low quality but used for posts. The bark serves as a mordant for dyes and is employed to relieve eye inflammation. The demand for the processed products is low and the prospect for the plant to be commercialized is currently very low. In Malaysia, the plant is cultivated only as a garden plant or in the orchards. No plantation is being largely developed to cultivate the plant due to its low commercial demand. However, with more upcoming researches on the ability of Rambai stem to be a promising alternative biosorbent generated, more values can be added to the plant and thus economically commercialized the plant. This is an advantage to Malaysia since the plant is originated and cultivated mostly in Southeast Asia region.

1.1.5 Heavy metal of concern: Nickel (Ni)

The metals of concern in creating pollution in water often include lead, chromium, uranium, selenium, zinc, arsenic, cadmium, copper and many others. The metals of concern for biosorption studies is dependent on the angle of interest and the impact of different metals, on the basis of which they would be divided into four major categories: (i) toxic heavy metals (ii) strategic metals (iii) precious metals and (iv) radio nuclides. In terms of environmental threats, it is mainly categories (i) and (iv) that are of interest for removal from the environment and/or from point source effluent discharges.

Nickel is an reactive element with the density of 8.908 g/cm^3 . Nickel is used in many industrial and consumer products, including stainless steel, magnets, coinage, and special alloys. It is also used for plating and as a green tint in glass. Nickel is pre-eminently an alloy metal, and its chief use is in the nickel steels and nickel cast irons, of which there are innumerable varieties. Nickel (III) oxide is used as the cathode in many rechargeable batteries, including nickel-cadmium, nickel-iron nickel hydrogen and nickel-metal hydride, and used by certain manufacturers in Li-ion batteries.

1.1.6 Health Hazard Analysis of Nickel

Heavy metals are natural components that cannot be degraded or destroyed. To a small extent they enter human bodies via food, drinking water and air. As trace elements, some heavy metals (e.g. copper, selenium, zinc) are essential to maintain the metabolism of the human body. However, at higher concentrations they can lead to poisoning. Heavy metal poisoning could result, for instance, from drinking-water contamination (e.g. lead pipes), high ambient air concentrations near emission sources.

Heavy metals are dangerous because they tend to bioaccumulate. Bioaccumulation means an ncrease in the concentration of a chemical in a biological organism over time, compared to the hemical's concentration in the environment. Compounds accumulate in living things any time ney are taken up and stored faster than they are broken down (metabolized) or excreted.

leavy metals can enter a water supply by industrial and consumer waste, or even from acidic in breaking down soils and releasing heavy metals into streams, lakes, rivers, and groundwater. ow we are going to describe the kinds of heavy metals, their dangerous levels and the effects of ese heavy metals to human health and environment.
ie most pollutans heavy metals are Lead, Cadmium, Copper, Chromium, Selenium and ercury.

mmon metals and their sources

- Lead: leaded gasoline, tire wear, lubricating oil and grease, bearing wear
- Zinc: tire wear, motor oil, grease, brake emissions, corrosion of galvanized parts
- Iron: auto body rust, engine parts

- Copper: bearing wear, engine parts, brake emissions
- Cadmium: tire wear, fuel burning, batteries
- Chromium: air conditioning coolants, engine parts, brake emissions
- **Nickel: diesel fuel and gasoline, lubricating oil, brake emissions**
- Aluminum: auto body corrosion
-

Small amounts of **Nickel** are needed by the human body to produce red blood cells, however, in excessive amounts, can become mildly toxic. Short-term overexposure to nickel is not known to cause any health problems, but long-term exposure can cause decreased body weight, heart and liver damage, and skin irritation. The EPA does not currently regulate nickel levels in drinking water. Nickel can accumulate in aquatic life, but its presence is not magnified along food chains.

1.2 PROBLEM IDENTIFICATION AND PROJECT SIGNIFICANCE

1.2.1 Problem Identification

There are many methods being incorporated in effort of reducing the heavy metals in wastewater. The well-established methods such as chemical precipitation or liming, ion exchange and activated carbon adsorption have well accepted in many large chemical industries.

However, these methods are also associated with other problems that require proper justifications. Chemical precipitation or liming is often associated with the production of a huge amount of sludge containing toxic compounds. The adsorption process using activated carbon is perhaps a better solution but due to the high selectivity and high cost associated with the methods, the interest had reduced significantly especially among those industries that often associated with lower amount of heavy metals to be treated.

Thus, the use of agricultural wastes or the biomass products would be a proper solution. The adsorbents develop from the sources can reduce the need for disposing the waste products from the agricultural industries and at the same time countering the disadvantages associated with other types of method to remove heavy metals, such as:

- ✓ Incomplete metal removal
- ✓ High reagent requirement
- ✓ Generation of toxic sludge
- ✓ Require careful disposal
- ✓ High operating cost
- ✓ Ineffectiveness in treating a large volume of effluents with low concentration of pollutants.

1.2.2 The significance of the Research Project

There are many researches and studies being develop on the subject of biological adsorption. However, this research on biosorption using Rambai stem is the first being conducted in this area and thus the results and development process will be subjected to many ambiguities and uncertainty. However, it is hoped that after the completion of the project, more researches and studies will be continued on this idea so that another option for heavy metals removal method can be developed.

This research paper will be concerned more on the feasibility of utilizing Rambai stem as one of alternatives in removing heavy metals in wastewater. The results and the findings of this research paper can perhaps provide a platform for the future research on this potential adsorbent. However, it is also expected that there will be many improvements shall be done in the methodology of conducting the research as well as the interpretation of the results and the findings from this paper for further development on the adsorbent.

1.3 OBJECTIVES AND SCOPE OF STUDY

1.3.1 The Objectives

The goal of the proposed project is to study preparation and use of low cost, organic agricultural material, which is Rambai stem as the biosorbent for removal of nickel from water. In more details, the project aims at the following targets:

1. Selection and preparation of the biosorbents
2. Conducting batch experiments to investigate the feasibility of Rambai stem as an adsorbent
3. Determination of the optimum condition for the adsorption process (pH, dosage of adsorbent and initial concentration of heavy metals) through sets of experiments.

1.3.2 Scope of Study

This research project will provide a review on the biosorption techniques using Rambai stem in removing heavy metals from water and how it can be a successful alternative in removing heavy metals with cost effective and eco-friendly ways, while other conventional techniques fail.

The main intention of the project is to identify the capability of Rambai stem to be an alternative biosorbent to remove heavy metals. The experiments conducted to test the optimum conditions for the adsorption process may be in general.

The adsorbed metals from aqueous solution may be further desorbed from the adsorbent using methods such as thermal regeneration or others before the metals adsorbed can be treated further to be used or disposed in a proper manner (such that reducing impacts to environment). However, for the simplicity of the project, those methods of desorbing the metals will not be put into discussion.

The project focuses on the ability of the biosorbent to adsorbed heavy metals from aqueous solution and the optimum condition for the adsorption process to take place, rather than identifying the properties of the adsorbent that make it suitable for the purpose. In other words, the project does not concern more on the characteristic posses by the adsorbent, such as the porosity, structure of the adsorbent molecules, pore volume and others associated.

The findings from the studies can be further used as a platform for future development and improvement. Due to time constraint, this research project does not aimed to provide all the necessary information in preparing Rambai stem to be a well-established type of adsorbent for industrial use. The recommendation part in the report will outline several identified improvements that can be done for the study.

Due to the unavailability of previous research on Rambai stem as an adsorbent for removing heavy metals, the results and the methodology used in the researched will be compared and based on several similar research papers featuring the types of adsorbent with almost similar properties. Though there may not be a precise explanation of the adsorption mechanism and reasons underlying the findings, this general comparative discussion of the results would hopefully give some general idea of the performance of Rambai stem as heavy metal adsorbents.

CHAPTER 2

LITERATURE REVIEWS

Many researches and studies had been done on biological type of adsorbent. There are many sources for biological adsorbent, from animal resources (animal bones, hair and feather) to biomass products such as microbial biomass and agricultural wastes. The examples of microbial biomass that are currently in great interest to be developed as biosorbent are seaweed, fungi and yeasts, certain bacteria and many others in the same group.

A few examples of literature reviews featuring various types of biomass from agricultural waste and microbial biomass in removing heavy metals are referred during research period of the project. These literatures would be able to help in explaining the results that are obtained by using Rambai stem as the heavy metals adsorbent since there is no such project involving the use of Rambai stem as the adsorbent under studies. Below are some of the details extracted from the literature reviews.

In a research done in 2006 by Muhammad Nadeem Zafar, Raziya Nadeem, Muhammad Asif Hanif (University of the Punjab, Pakistan), the removal of Nickel from aqueous medium using protonated rice bran was investigated. The adsorption characteristics of nickel on protonated rice bran were evaluated as a function of pH, biosorbent size, biosorbent dosage, initial concentration of nickel and time.

For the pH effect on the adsorption process, the protonated rice bran displayed up to 102 mg/g of the nickel binding capacity at pH 6 and getting decrease in uptake capacity at lower pH values. The percentage removal of nickel was at a maximum value for 0.25 g of biosorbent dose and 0.25 mm biosorbent size. At the optimal conditions, metal ion uptake was increased as the initial metal ion concentration increased up to 100 mg/L. Kinetic and isotherm experiments were carried out at the optimal pH 6.0 for nickel. The metal removal rate was rapid, with 57% of the total adsorption taking place within 15–30 min. The Freundlich and Langmuir models were used to describe the uptake of nickel on protonated rice bran. The Langmuir and Freundlich model parameters were evaluated. The equilibrium adsorption data was better fitted to Langmuir adsorption isotherm model.

In another research paper from Dr Nasim Ahmad Khan (Aligarh Muslim University, India) and Mr Hapsah Mohamad (Universiti Malaya, Malaysia) in 2007 features the studies of sugarcane bagasse as an adsorbent in removing Chromium from the synthetic wastewater. The effects of various parameters such as a adsorbent dosage , solution pH, contact time, and initial Cr (VI) concentration on adsorption efficiency were studied trough extensive batch experiments.

From the studies, it is found out that the removal of heavy metal was effective at low pH values and low Chromium concentration. The Cr (VI) removal efficiency was found to be 70.2% at initial Chromium concentration of 10 mg/L (10 ppm) at pH 1 and at 4 contact hour. An adsorbent dosage of 7 g/L was sufficient for the removal of Cr (VI) from wastewater.

Another research was done by Guiqiu Chen *et al* (Hunan University) in 2007 to investigate the removal of Cadmium from simulated wastewater using the biomass product of *Lentinus edodes*. *Lentinus edodes* is a byproduct of brown-rot fungus, a kind of agricultural waste. Like the previous research done, the sorption conditions such as pH, adsorbent dosage and initial concentration of the metals solution were investigated.

The results from series of experiment conducted during the studies had revealed that nearly no sorption occurred when the pH value was lower than 2.5. The higher adsorption capacity occurred at the weak acid range which is around pH 6.5. The sorption showed that the maximum biosorption capacity occurred at the biomass dosage of 1 g/L. The biosorption capacity was decrease with the increase in biosorption quantity. Among the three adsorption isotherm being investigated, Freundlich was found to be the most suitable for the adsorption process involving *Lentinus edodes.*

Mariam Kazimepour *et al* (Islamic Azad University, Iran), in 2007 have investigated the potential of carbon developed from walnut, hazelnut, almond, pistachio shell and apricot stone in removing Lead, Cadmium, Zinc and Copper from industrial wastewater. Removal efficiency was optimized regarding to the initial pH, flowrate and dose of adsorbent.

The maximum removal occurred at pH 6 -10, flowrate of 3 mL/min and 0.1 g of the adsorbent. Capacity of carbon sources for removing cations will be considerably decreased in the following times of passing through them. Results showed that the cations studied significantly can be removed by the carbon sources. Efficiency of carbon to remove the cations from real wastewater produced by copper industries was also studied. Findings showed that not only these cations can be removed considerably by the carbon sources noted above, but also removing efficiency are much more in the real samples. These results were in adoption to those obtained by standards mixture synthetic wastewater.

From those researches done, it can be extracted that the conditions for the adsorption process involving different types of adsorbent are quite different from one another. This may be caused by the difference in the properties possessed by each type of biosorbent. The biosorption process associated with certain metals also depends on various factors such as the number of sites in the biosorbent materials, the accessibility of the sites, the chemical state of the sites and affinity between sites and metals (Regine and Volesky, 2000).

From a 2007 research paper by Hima Kanika Alluri *et al* (Koneru Lakshmaiah College of Engineering, India), it is outlined that there are various types of biomass to be selected as the adsorbent in removing heavy metals in wastewater. It can come from, activated sludge or fermentation waste from industries, living organisms (yeast, fungi, algae, bacteria etc.) and agricultural products such as wool, rice, straw, coconut husks, walnut skin and many others.

According to the researches, those microbial biomass sources for adsorption need to be further treated before able to be used as adsorbent. The pre-treating and immobilization procedures of the microbial biomass is needed for extending the shelf-life of the microbial biomass and because of the properties of the microbial biomass that is low density, poor mechanical strength and little rigidity. As for the free biomass such as agricultural wastes, no such process needed. However, in a long run, those types of adsorbent are easily biodegradable and thus making it less favourable in a long term experimental procedures.

Taking those factors into account, the investigation of Rambai stem as biosorbent in removing heavy metals shall be conducted in a way that reduce the possibility of biodegradability of the adsorbent. These types of adsorbent are also suitable for the project, considering the limited time given for the project completion.

CHAPTER 3

METHODOLOGY

From the researches and literature review, the methodology for conducting experiments for biosorption process is identified. These are based on the existing methods utilized by other researches in developing biosorbents for heavy metals removal, using other types of agricultural materials. Below are the details of the methods used in the experimental part of the projects.

3.1 EXPERIMENTAL PROCEDURES:

3.1.1 Preparation of the adsorbent (Rambai stem) and solution of adsorbate (Ni).

i. The adsorbent

The Rambai stem used in this study was obtained from a local orchard. The collected material is extensively washed with tap water to remove soil and dust and other growing organisms on the surface of the stems before being cut into smaller pieces. The smaller fragments of the stem were then dried in a hot air convection oven overnight. After that, the stems were soaked in hot water at the temperature of 70°C for half an hour before being rinsed with distilled water until the surface of the stem clean. Then, the stems were dried again at the temperature of 105°C, for a couple of days. This is to ensure that the stems are completely dried and easy to be crushed later. The dried materials were milled into small particles using a mortar and pestle. The treated adsorbent will be stored in a closed container for use.

ii. The adsorbate

The metal selected to be used in this project is nickel. A solutions containing 1000 ppm of nickel were prepared using nickel nitrate solid. All the respective concentrations that will be used in the experiments will be prepared using the 1000 ppm solution. Nitric acid (HNO3) 0.01 M and Sodium Hydroxide (NaOH) 0.01 M were also prepared for the pH experiment. The samples for the related calculation are as in Appendix 1.

3.1.2 Isotherm Experiment

Isotherm experiment was conducted to describe the amount of adsorbate on the adsorbent as a function of concentration of adsorbate at constant temperature.

1. For this purpose, a varied amount of adsorbate (nickel) concentrations were introduced; 10, 20, 30, 40 and 50 ppm. Each at pH 5.
2. The adsorbent (Rambai stem granules) was weighted and taken as 0.1 g for each sampling set.
3. The adsorbent was mixed with the 100 ml of solutions, each with different concentration of Nickel.
4. The sample bottles were shook on the automatic shaker at 160 rpm at room temperature.
5. The sampling was done every 1 hour for 5 hours of total contact time.
6. A 10 ml sample of each set of sample bottle was taken and filtered using filter paper to separate the adsorbent granules.
7. The samples were then analyzed using Atomic Adsorption Spectroscopy.

Due to unavailability of AAS analyzer for a period of time, the samples need to be stored. Therefore, a few drops of diluted 1% of HNO_3 acid were introduced into the samples to avoid the growth of fungal or other foreign organism.

3.1.3 Kinetics Experiments

The influences of several operational parameters such as pH, initial concentration of heavy metals as well as the dosage of adsorbent (Rambai stems) on the adsorption process are also included in the study.

i. **Effect of initial concentration**

For the initial concentration experiment, the procedures involve are similar with the isotherm experiment as mentioned above. For this purpose, the concentration of the adsorbate (Nickel) will be varied as 10, 20, 30, 40 and 50 ppm. The preparation of the solution concentration was done using the 1000 ppm of Nickel solution. For the simplicity of the experiment, the sampling time for the experiment will be taken as the equilibrium time obtained from isotherm experiment, which is after 5 hours of contact time. Other parameters such as pH and adsorbent dosage are kept as constant which are 5 and 0.1 gram, respectively. The temperature and the speed of the shaker are set as room temperature and 160 rpm, respectively. The rest of steps for the experiment are similar with the isotherm experiment.

ii. **Effect of pH**

To investigate the effect of pH, other parameters such as initial concentration of adsorbate (taken to be 30 ppm) and adsorbent dosage (0.1 g) are kept constant throughout the experiment. The pH value range for the nickel solution were varied according to low pH range (pH 2-3), medium pH range (4-5) and high pH range of 6-7. The pH values were determined by using pH paper and therefore, the values for each pH set is allowed to be varied within the range. The Sodium Hydroxide solution of 0.01 ppm and Hydrochloric acid were used to increase and decrease the pH value, respectively. The sampling procedure was done after the 4^{th} hour of contact time. This is to allow the pH of the solution to take effect. The rest of the steps were similar with the isotherm experiment (step 6 and 7 in previous page).

iii. Effect of dosage

As for the effect of dosage of adsorbent, the values for the dosage were varied as 0.05 g, 0.1 g and 0.2 g. Other parameters such as pH and initial adsorbate concentration are kept constant as 5 and 30 ppm, respectively. The experiment conditions such as the temperature and shaker speed are as the other two experiments. The sampling time for the experiment was also varied as 5, 15,30,45 and 60 min. The rest of the experiment steps are as step 6 and 7 in the isotherm experiment.

3.2 TOOLS / EQUIPMENT USED

Atomic absorption spectroscopy is a technique for determining the concentration of a particular metal element in a sample. Atomic absorption spectroscopy can be used to analyze the concentration of over 62 different metals in a solution. The technique makes use of absorption spectrometry to assess the concentration of an analyte in a sample. It relies therefore heavily on Beer-Lambert law.

In short, the electrons of the atoms in the atomizer can be promoted to higher orbitals for an instant by absorbing a set quantity of energy (i.e. light of a given wavelength). This amount of energy (or wavelength) is specific to a particular electron transition in a particular element, and in general, each wavelength corresponds to only one element. This gives the technique its elemental selectivity.

As the quantity of energy (the power) put into the flame is known, and the quantity remaining at the other side (at the detector) can be measured, it is possible, from Beer-Lambert law, to calculate how many of these transitions took place, and thus get a signal that is proportional to the concentration of the element being measured.

3.3 BATCH EXPERIMENT

The batch experiment will be carried out in 100 ml capacity plastic sample bottle. The initial sample of 10 ml of the mixture of heavy metal solution together with the adsorbent dosage will be taken from the sample bottle for sampling purpose before the experiment is conducted. The rest of the mixture will be left on the shaker at room temperature within respective time intervals for each experiment. The content will be centrifuged at 160 rpm. The sample of 10 ml will be taken during sampling period and filtered using filter paper before being determined using spectrometer. The uptake of metal ions on the adsorbent at different initial concentration of feed solution was calculated.

3.4 METAL UPTAKE EVALUATION

The metal uptake capacity from the data obtained would be evaluated using the formula (Volesky, 1999, p.136):

$$\text{Metal uptake, q} = V[L]x\frac{(C_i - C_f)[mg/L]}{S[g]} \quad \text{..............................} \textbf{Equation 1}$$

Where,

V = Volume of metal solution contacted with the sorbent.

C_I & C_f = Initial and equilibrium concentration of the metal in the solution

S = Amount of sorbent (Rambai stem granules) on dry basis.

The metal uptake could also be analysed by calculating the metal uptake rate as defined by the equation,

$$\text{Percentage adsorbed} = \frac{C_i - C_f}{C_i} \times 100\% \quad \text{..............................} \textbf{Equation 2}$$

CHAPTER 4

RESULT AND DISCUSSION

The results obtained for each set of experiment are tabulated in tables presented below and then plotted as graphs that followed. The discussion of the results shall be based on the percentage of adsorption according to each set of experiment.

4.1 RESULT

4.1.1 Isotherm Experiment

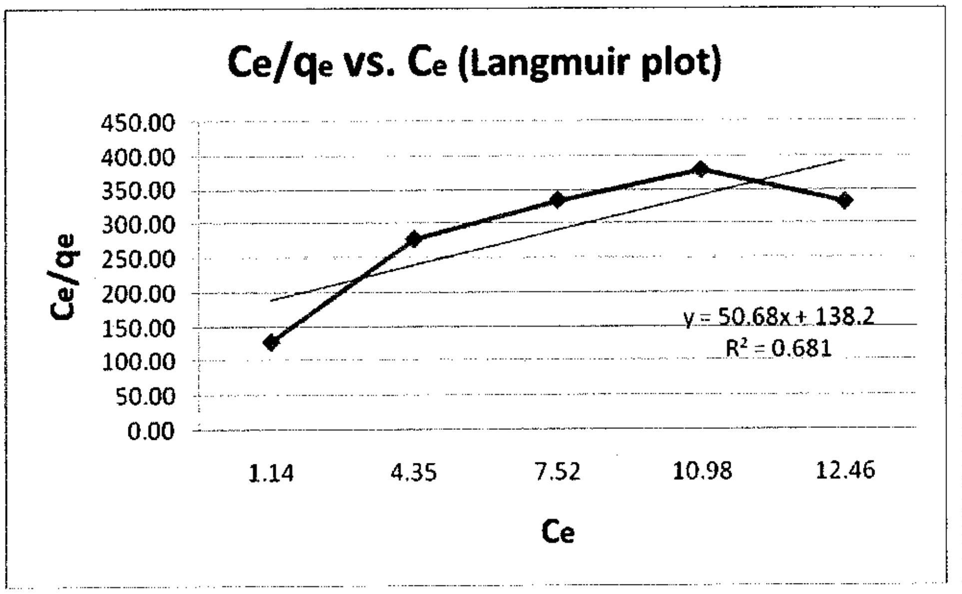
For adsorption isotherm determination, the data for the plot will be taken from the initial concentration experiment at the equilibrium, which is at 5 hours of contact time. The data are tabulated as below:

initial Concentration (Ci)	Amount of Adsorbent (Rambai stem), g	Concentration Remaining at equilibrium (Ce)	Concentration adsorbed	Amount of metal Adsorbed (mg), qe	ce/qe (mg/mg)	ln qe	ln Ce
10	0.1	1.14	8.86	0.0089	128.67	-4.73	0.13
20	0.1	4.35	15.65	0.0157	277.96	-4.16	1.47
30	0.1	7.52	22.48	0.0225	334.52	-3.80	2.02
40	0.1	10.98	29.02	0.0290	378.36	-3.54	2.40
50	0.1	12.46	37.54	0.0375	331.91	-3.28	2.52

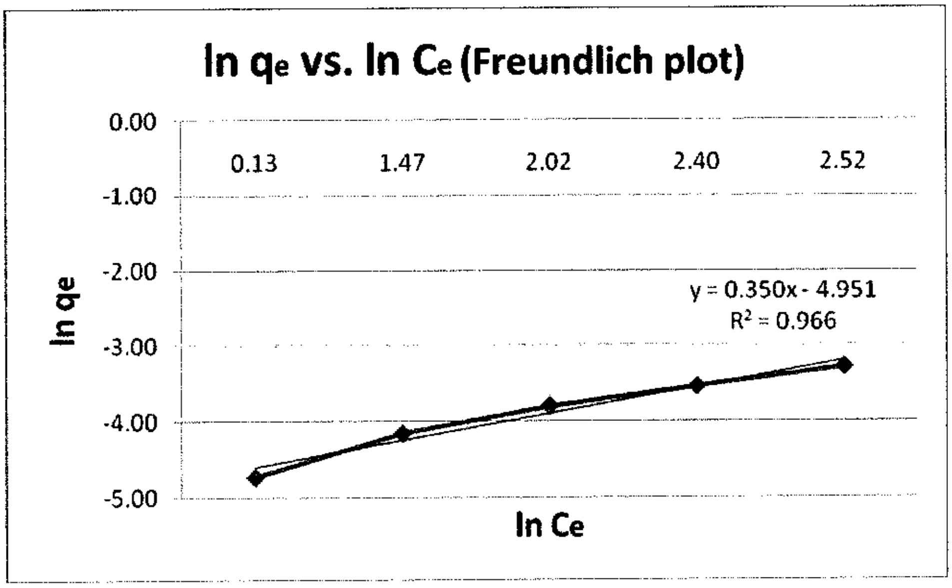
Table 4.1: Adsorbed metals at equilibrium, at 5 hours of contact time

Both Langmuir and Freundlich isotherms are plotted using the data as tabulated above and the plots are as shown in the next page:

i. **Langmuir plot:**



ii. **Freundlich plot**



4.1.2 Kinetic Experiment: Effect of Initial Concentration

For the effect of initial concentration experiment, the pH and dosage values for each sample were kept constant. The batch experiment was conducted with varied initial concentration.

Table 4.2: Concentration of remaining metal in the solution for different initial concentration

Contact time (hour)	Concentration of metal remains in the sample (ppm)				
	Initial conc 10 ppm	Initial conc 20 ppm	Initial conc 30 ppm	Initial conc 40 ppm	Initial conc 50 ppm
0	10	20	30	40	50
1	2.05	4.98	7.96	11.14	12.57
2	1.86	4.48	7.68	11.06	12.52
3	1.63	4.41	7.61	11.02	12.49
4	1.46	4.37	7.56	10.99	12.47
5	1.14	4.35	7.52	10.98	12.46

Figure 4.1: Concentration of remaining Nickel for different initial concentration of Nickel solution

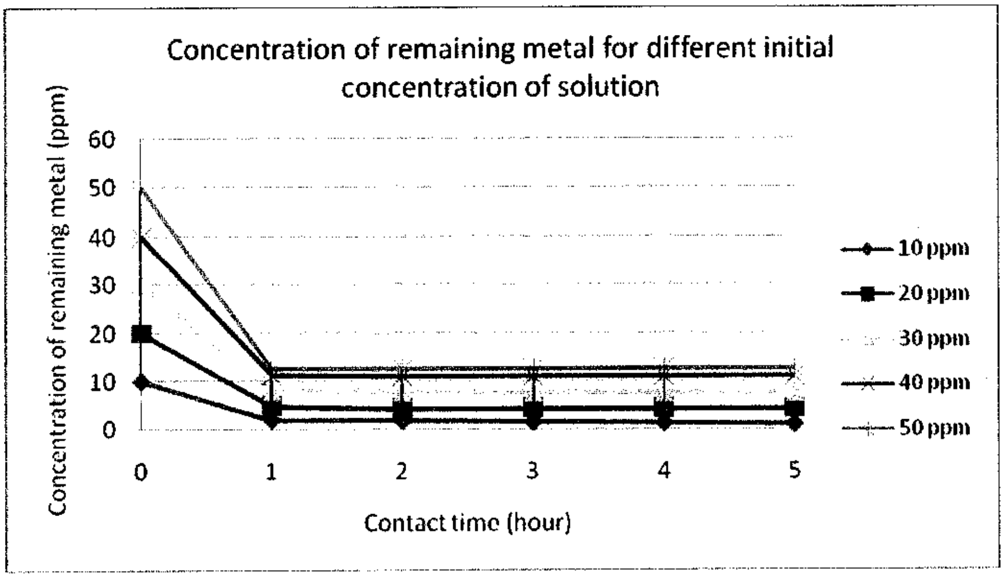
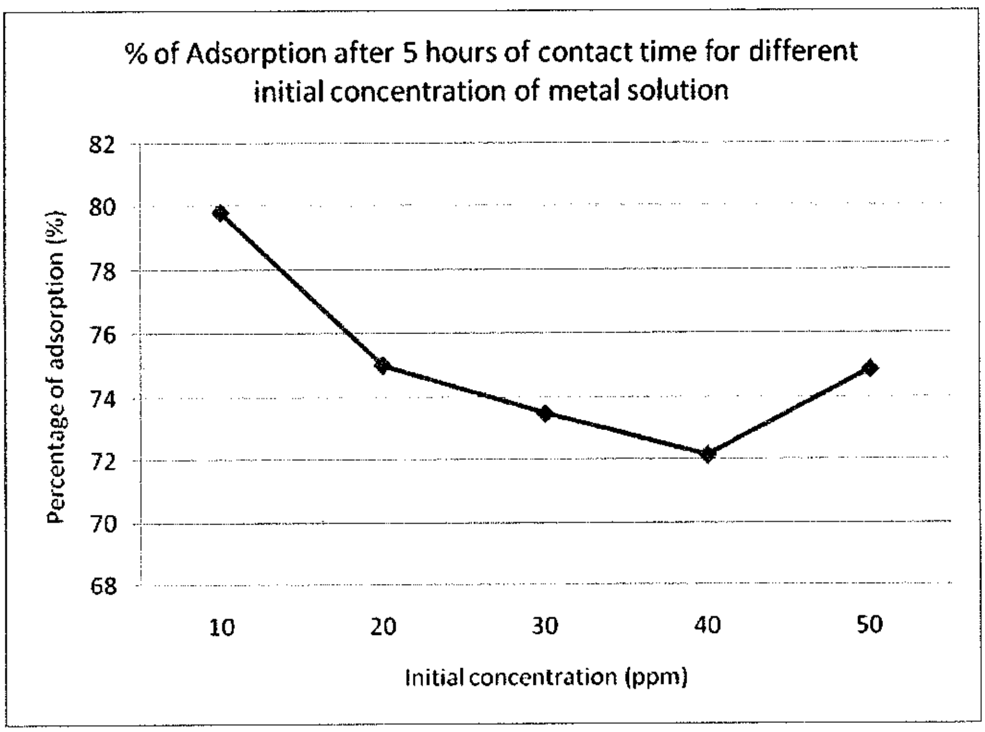


Table 4.3: Total concentration of adsorbed metal in the solution after 5 hours of contact time for different initial concentration and the removal percentage

Initial Concentration (ppm)	Total Adsorbed metal after 5 hours contact time (ppm)	% of Adsorption
10	8.89	79.8
20	15.65	75
30	22.46	73.47
40	29.02	72.15
50	36.04	74.86

Figure 4.2: Percentage of removal vs. different initial concentration



4.1.3 Kinetics Experiment: Effect of Adsorbent Dosage

Table 4.4: Concentration of remaining metal in the solution (Nickel 30 ppm) for different dosage of adsorbent (*Rambai* stem)

	Concentration of Metal remains in the sample (ppm)		
Contact time (min)	Dosage (0.05 g)	Dosage (0.1 g)	Dosage (0.2 g)
0	30	30	30
5	15.4	14.53	13.11
15	14.7	14.31	13.02
30	14.7	13.99	13
45	14.17	13.98	12.82
60	13.96	13.77	12.6

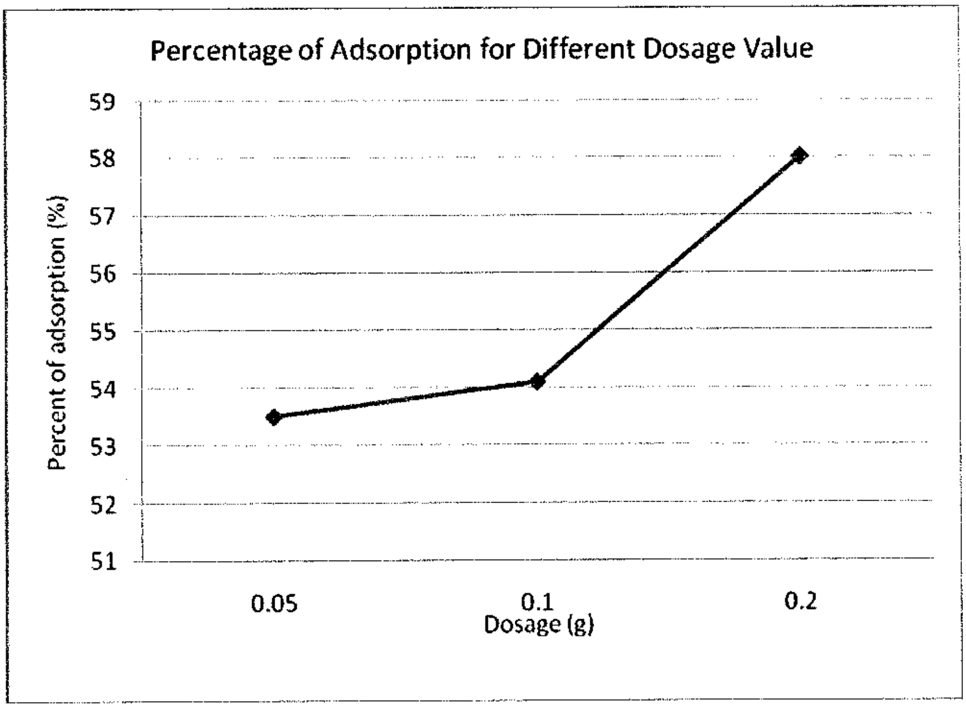
Table 4.5: Concentration of metal adsorbed from the solution (initial concentration: Nickel 30 ppm)

	Concentration of Metal Adsorbed (ppm)		
Contact time (min)	Dosage of 0.05 g	Dosage of 0.1 g	Dosage of 0.2 g
0	30	30	30
5	14.6	15.47	16.89
15	0.7	0.22	0.09
30	0	0.32	0.02
45	0.53	0.01	0.18
60	0.21	0.21	0.22
Total adsorbed	16.04	16.23	17.4

Table 4.6: Total concentration of adsorbed metal in the solution after 1 hour of contact time for different dosage and the removal percentage.

Dosage (g)	Total Concentration of metal adsorbed after 1 hour	% of adsorption
0.05	16.04	53.5
0.1	16.23	54.1
0.2	17.4	58

Figure 4.3: Graph of percentage of removal vs. different dosage value



4.1.4 Kinetics Experiment: Effect of pH

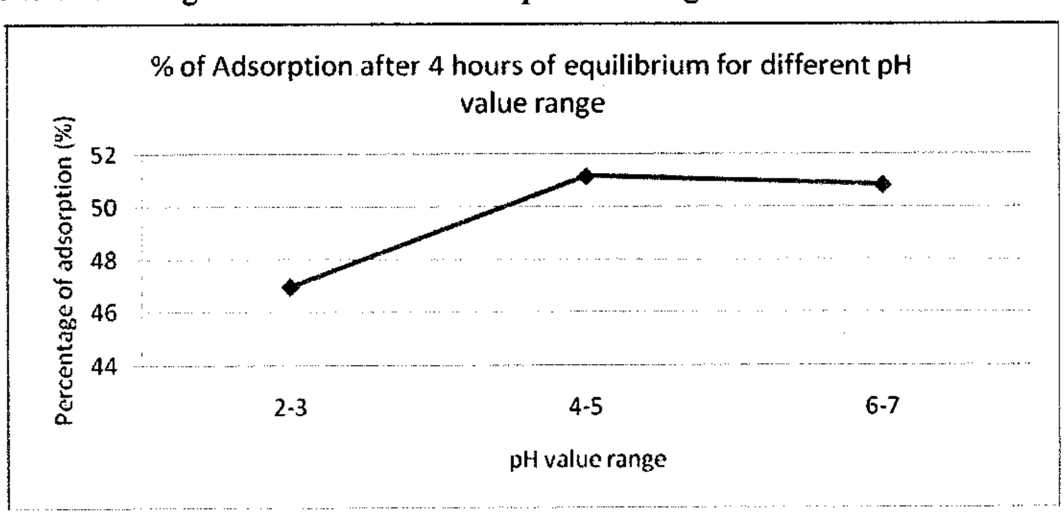
Table 4.7: Concentration of remaining metal in the solution of Nickel 30 ppm, 0.1 g of adsorbent (*Rambai* stem)

	Concentration of Metal Remaining in the solution	
pH Value	For 3 Hours of equilibrium	For 4 Hours of equilibrium
2-3	16.27	15.91
4-5	15.13	14.65
6-7	15.4	14.75

Table 4.8: Total concentration of adsorbed metal in the solution of Nickel 30 ppm, 0.1 g of adsorbent (*Rambai* stem) and the percentage of adsorption.

Concentration of Adsorbed metal from solution of Nickel 30 ppm, 0.1 g of adsorbent		
pH Value	Total Adsorbed Metal After 4 Hours of Equilibrium (ppm)	% of Adsorbed
2-3	14.09	46.97
4-5	15.35	51.17
6-7	15.25	50.83

Figure 4.4: Percentage of removal vs. different pH value range



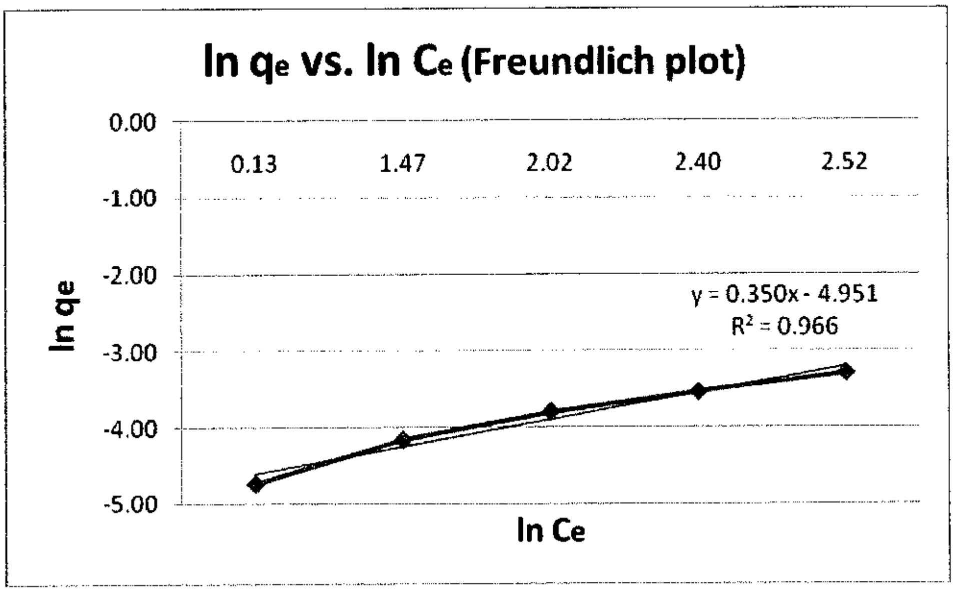
4.2 DISCUSSION

4.2.1 Isotherm Experiment

From the data obtained, the plot of both adsorption isotherm, Langmuir and Freundlich were done. From this, it can be observed that the data fitted Freundlich plot very well with associated regression coefficient of 0.966.

From this result, it can be concluded that the adsorption using Rambai stem does follow a multilayer or heterogeneous surface of adsorption. The adsorption process may have altered the surface of the adsorbent and thus, further allow for another layer of adsorption to take place.

Figure 4.5: Freundlich plot



4.2.2 Kinetic Experiment: Effect of Initial Concentration

The effect of initial concentration was carried out in a batch experiment where the dosage of adsorbent and pH value are set and kept constant at 0.1 g and 5, respectively. The sampling procedure was done every 1 hour up to 5 hours for the total contact time. The amount of adsorbed metals was then summed up and was taken to be the final concentration.

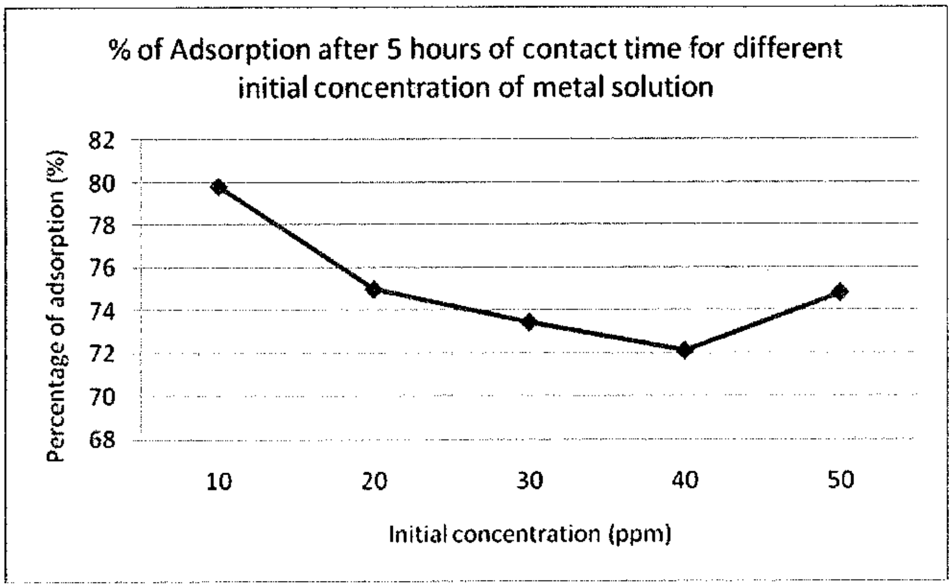


Figure 4.6 : Removal percentage (R%) of Nickel for different initial concentration after 5 hours

The graph indicates the removal percentage (percentage of adsorption) for different initial concentration. The trend of the graph showed a decrease in percentage of removal as the initial concentration increase from 10 to 40 ppm. This signifies that the adsorption is high at lower initial concentration of metal solution. The highest removal percentage was found at initial concentration of 10 ppm.

At 50 ppm, however, the adsorption again increased. This may be due to errors done during the preparation of the sample. Based on the previous similar study, it is expected that the percentage of removal of heavy metals at lower initial concentration should be higher than the higher initial concentration. This is due to the fact that the smaller dosage of adsorbent (that was kept constant throughout the experiment) cannot cater for a huge amount of metal within the solution. The limited number of available sites for adsorption may be exhausted even when the concentration of metal in the solution is still at a high amount.

It is also found out that the highest adsorption values occurred at the first 1 hour of contact time compared to the rest of contact times for the experiment. This signifies that the adsorption using Rambai stem is considerably rapid.

4.2.3 Kinetics Experiment: Effect of Adsorbent Dosage

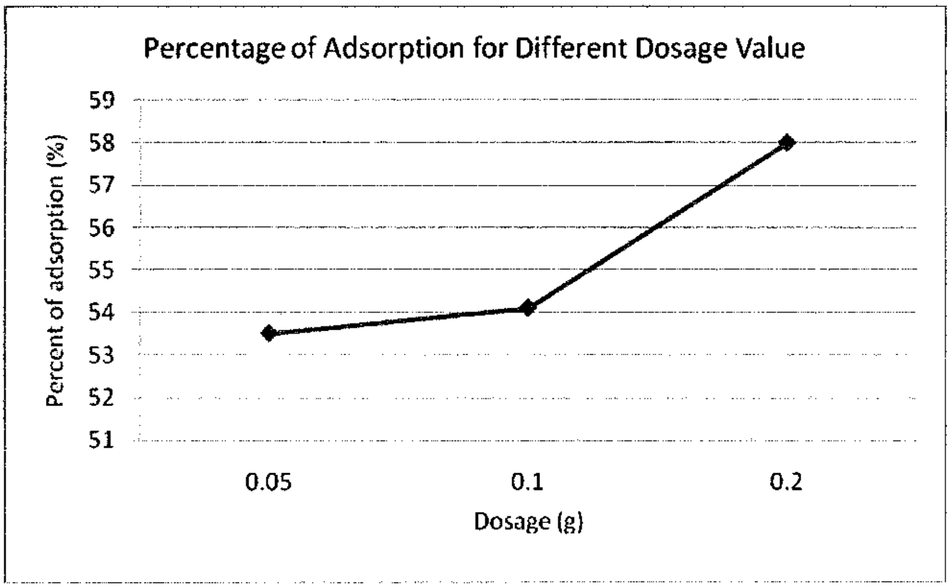


Figure 4.7: Removal percentage (R%) of Nickel for different adsorbent dosage after 1 hour of contact time

For the effect of adsorbent dosage, the batch experiment was conducted by maintaining the value of other parameters, the pH and initial concentration at 5 and 30 ppm, respectively. The sampling procedure was done every 5, 15, 30, 45 and 60 minutes for every set of batch experiment containing different amount of adsorbent dose. The amount of adsorbed metals was then summed up and was taken to be the final concentration, which is at 1 hour of contact time. The sampling time is taken to be within 1 hour only considering the result from the first experiment, which is the initial concentration experiment, where it signifies that the highest adsorption of heavy metal from solution occurred at the first hour of the contact time. Therefore by taking sample within the first hour on contact time can give clearer result and make it easy to be compared.

The graph indicates the removal percentage (percentage of adsorption) for different dose of adsorbent. The trend of the graph showed a increase in percentage of removal as the dosage increase from 0.05 to 0.2 g. This signifies that the adsorption is high at higher amount of adsorbent. The highest removal percentage was found at 0.2 g of adsorbent (Rambai stem).

This may be explained by the fact that the higher the amount of adsorbent available the higher the sites will be available for the adsorption of heavy metal to be taken place. It is also can be seen that difference between the removal percentage associated with 0.05 g and 0.1 g of adsorbent dosage is small. This may be due to the smaller difference in the amount of dosage of adsorbent used and thus the difference in binding sites available within the adsorbent to take up the metal in the solution is also small.

4.2.4 Kinetics Experiment: Effect of Metal Solution pH

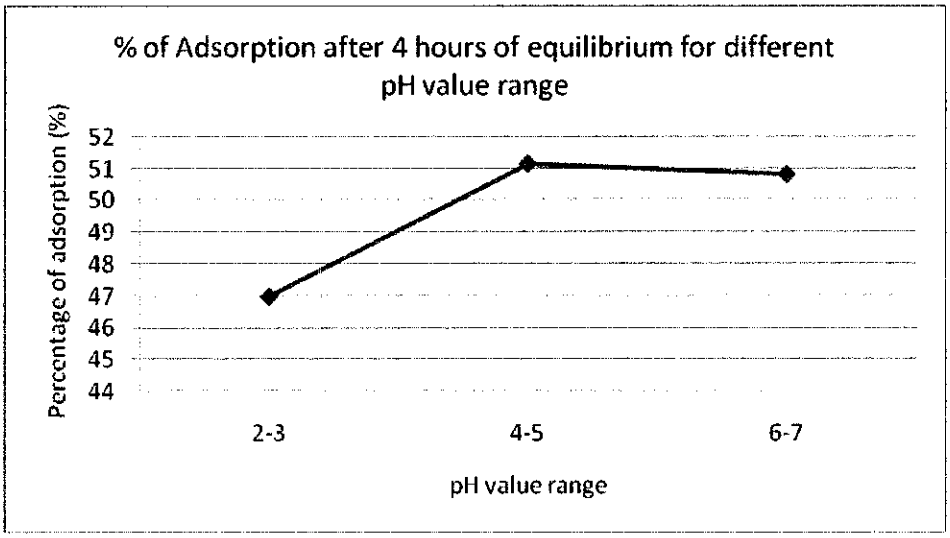


Figure 4.8: Removal percentage (R%) of Nickel for different adsorbent dosage after 1 hour of contact time

For the effect of metal solution pH, the batch experiment was conducted by maintaining the value of other parameters, the adsorbent dosage and initial concentration at 0.1g and 30 ppm, respectively. The sampling procedure was done at 3 and 4 hours of contact time for every set of batch experiment containing solutions with different value for pH. The amount of adsorbed metals was then summed up and was taken to be the final concentration, which is at 4 hour of contact time. The sampling time is taken to be at 3 and 4 hours to allow for the pH of the solution to be settled so that the effect can take place. However, for the simplicity of the discussion on the result, the results at 3 and 4 hours of contact time will be summed up and will be regarded as the final amount of adsorbed.

The pH for the experiment is divided into 3 condition; weak acid condition (pH ranges between 4 to5), strong acid condition (pH ranges between 2 to 3) and neutral condition (pH ranges between 6 to 7). The experiment was conducted in acidic range of pH for the solution considering the previous researches that indicate most of the adsorption process involving biological adsorbents was optimum within acidic range of pH. The pH values were taken to be in ranges since the experiment was done using basic apparatus which is pH paper instead of pH meter that need proper analytical procedure to determine the exact pH values.

As for the graph, it indicates the removal percentage (percentage of adsorption) for different pH range of the adsorbate solution. The trend of the graph showed that at the strong acid condition (pH range of 2-3), the adsorption is the lowest while the highest adsorption of Nickel is found out to be at the weak acid condition with associated pH range between 4-6. For the neutral condition, the graph showed a slight decrease. If the experiment is continued with pH range in alkaline condition, the result will most probably drop. This is based on the drop of adsorption percentage as the pH range getting closer to neutral range.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

As conclusion for this research, it is identified that Rambai stem is capable in removing heavy metal from aqueous solution by following the Freundlich adsorption isotherm. From series of experiments done to identify the condition for the optimum adsorption, the findings are as below:

1. The **pH range** that produce the highest percentage of adsorption is in **weak acid** (pH range between 4 to 5) when the dosage and initial concentration are 0.1 g and 30 ppm
2. The highest percentage of removal of Nickel from the solution is found out at **0.2 g of adsorbent dosage** when the pH is set at 5 and initial concentration of Nickel solution is 30 ppm
3. The highest percentage of adsorption is the highest when the **initial concentration of Nickel is at the lowest**, which is 10 ppm at the end of 5 hours of contact time, with adsorbent dosage of 0.1 g and pH of 5.

From the effect of initial concentration experiment, it is also found that the **adsorption process using Rambai stem is a rapid process** with the highest removal percentage occurring in the first hour of contact time. Also, based on the comparison between the adsorption using activated carbon obtained from other research paper and adsorption process using Rambai stem, it is found out that the adsorption using Rambai stem can be taken as **comparable with the result obtained using activated carbon**.

5.2 RECOMMENDATIONS

From the experiment conducted in the research project, it can be concluded that Rambai stem has the ability to become an adsorbent in removing heavy metal from aqueous solution. However, due to some constraints encountered during the project, some improvement is required in order to justify the result further. Some of the improvements that can be done are:

1. Further study on the ability of the adsorbent to remove other heavy metals such as Chromium, Mercury and etc.
2. Further review on the methodology and equipments used during the experimental part of the project to obtain more accurate results.
3. Further study on other parameters that may affect the result for the adsorption process using Rambai stem such as contact time, speed of agitating shaker and size of particles of adsorbent.
4. Further consideration on the HSE aspect of the project, since this research project is associated with various hazardous chemicals.

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APPENDIX

1) **Sample of calculation**

   i. Preparation of adsorbate solution,
   Example: Nickel solution from Nickel Nitrate Hexa-Hydrate, Ni $(NO_3)_2.6H_2O$

Molecular weight (MW) of Ni $(NO_3)_2.6H_2O$ = 290.83 g/gmol
Atomic weight (AW) of Nickel = 58.7 g/gmol

X (amount of absorbate in gram) *(AW of Nickel / MW of Nickel Nitrate) = 1
X* (58.7 / 290.83) = 1
X = **4.955 ≈ 5 grams**

*The 5 grams of Ni $(NO_3)_2.6H_2O$ will be diluted further with distilled water up until 1000 mL to produce 1000 ppm of Nickel solution.

   ii. Preparation of sampling concentration of the adsorbate
   Example: 5 ppm of Nickel solution (250 mL)

Using the formula, $M_1V_1 = M_2V_2$

*M = molar concentration, V = volume

$$M_1V_1 = M_2V_2$$
$$(1000)\,V_1 = (5)(250)$$
$$V_1 = \mathbf{1.25\ mL}$$

*Summary table indicating the value of volume used for each different concentration,

Solution Concentration (ppm)	Solution Volume (mL)
5	1.25
10	2.50
20	5.00
30	7.50
40	10.00
50	12.50

iii. Calculation for percentage of removal (R%)

$R (\%) = (C_i - C_f)/C_i \times 100$

Example: Taking the initial concentration of 10 ppm

$R (\%) = (10-8.89)/10 \times 100 =$ **79.8**

The example of calculated % of removal for **the effect of initial concentration** experiment:

Initial Concentration (ppm)	Total Adsorbed metal after 5 hours contact time (ppm)	% of Adsorption
10	8.89	79.8
20	15.65	75
30	22.46	73.47
40	29.02	72.15
50	36.04	74.86

2) Other Rambai stem characterization analysis

From the analysis performed as below, a general preview of the stem granules characteristics can be revealed. However, these analysis only cover a simple explanation on the characteristics of the stem. More details analysis shall be done in the future to investigate on the characteristics of the stem in order to clearly explain how these characteristics can affect the adsorption process using Rambai stem.

I. Fourier Transformation Infrared Spectrophometer (FTIR) analysis

The analysis using FTIR was done to identify the types of chemical bonds (functional groups) associated with Rambai stem granules. The organic compound, such as the Rambai stem has very rich, detailed spectra. As for most cases involving common materials, the spectrum of an unknown material can be identified by comparison to a library of known compounds. The details of the analysis result are as previewed below:

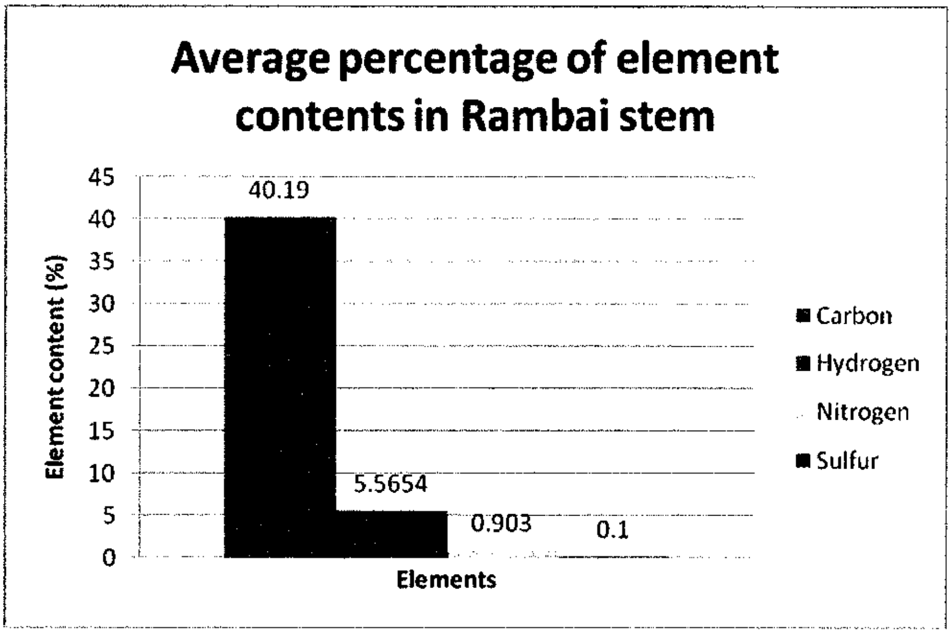
Peak (1/cm)	Interpretation		
	Functional group	Formula	Vibration mode
3500-2800	Hydroxyl	O-H	Stretching
2955-2922	Methylene	CH_2	Antisymmetrical stretching
1100-1060	Primary alcohol	C-O	Stretching
2170-1900	Carbonyl	C≡O	Stretching
1350-1310	Secondary amides	C-N	Stretching
1641	Cyclic	C=C	Stretching

* the plotted result from FTIR is as followed in the next page

II. Carbon Hydrogen Nitrogen Sulfur (CHNS) Determination

The test is done to identify the percentage of the element content in Rambai stem granules. The main elements being identified are carbon, hydrogen, nitrogen and sulfur. 5 samples are run and the average is taken from each sample data. Below are the details of the findings:

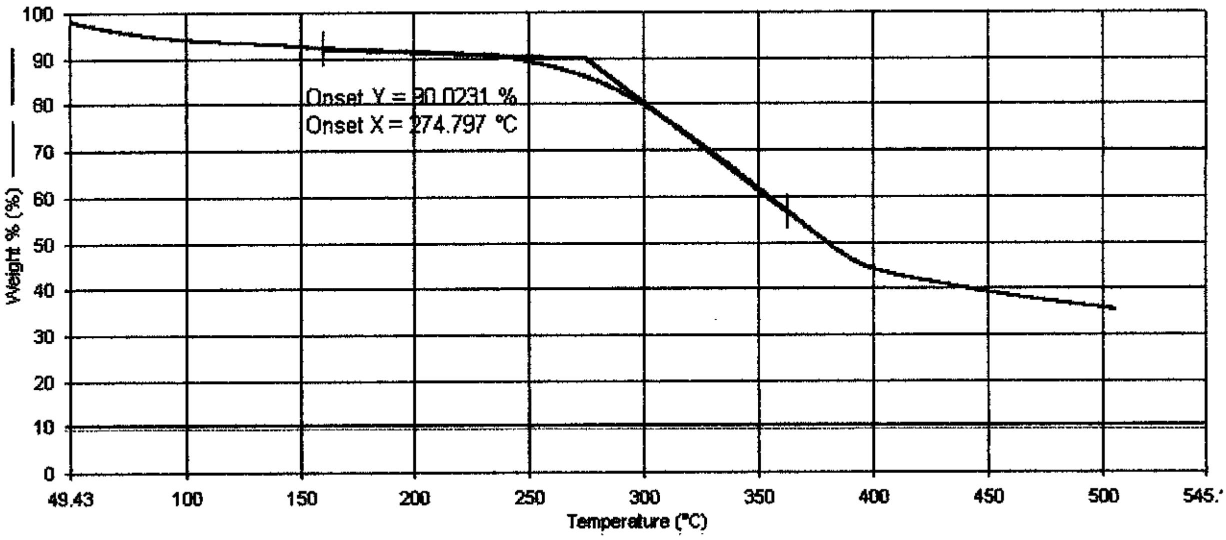
CHNS Determinator Result				
Percentage of elements (Carbon, Hydrogen, Nitrogen and Sulfur) content ,%				
Sample	Carbon	Hydrogen	Nitrogen	Sulfur
1	40.03	6.124	0.906	0.133
2	39.68	5.695	0.886	0.11
3	40.78	5.503	0.896	0.101
4	40.16	5.32	0.935	0.087
5	40.3	5.185	0.892	0.069
Average	40.19	5.5654	0.903	0.1



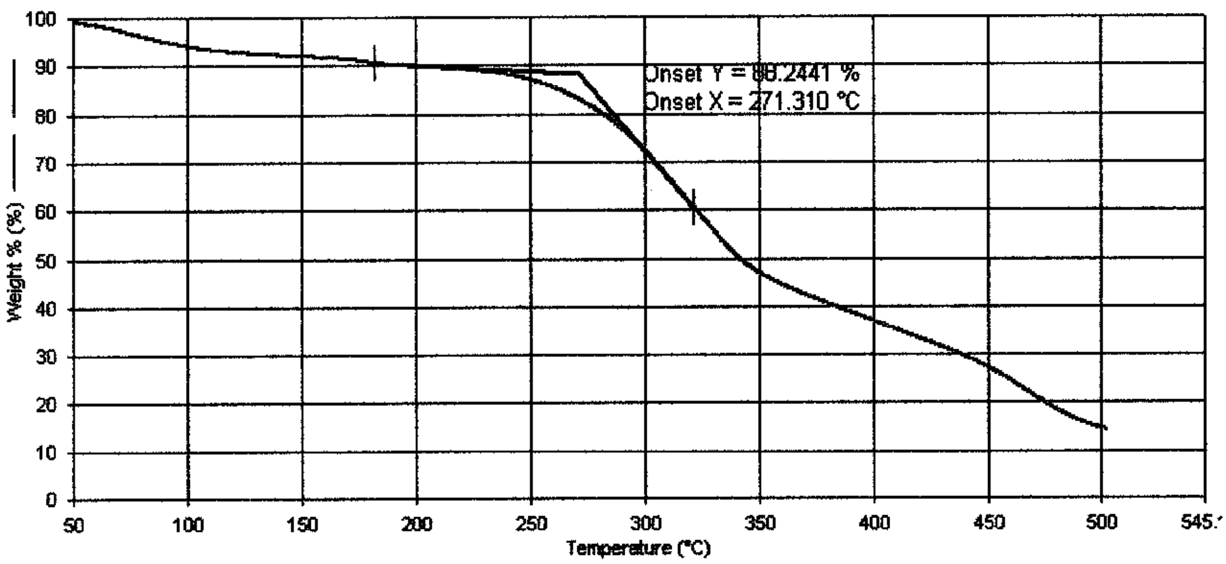
III. Thermal Gravitational Analysis

This is a simple analytical technique that measures the weight loss (or weight gain) of a material as a function of temperature. As materials are heated, they can loose weight from a simple process such as drying, or from chemical reactions that liberate gasses. Some materials can gain weight by reacting with the atmosphere in the testing environment. Since weight loss and gain are disruptive processes to the sample material or batch, therefore the knowledge of the magnitude and temperature range of those reactions is necessary. Below are the result obtained from the analysis with different heating rate of 10, 15 and 20 C/min. The rate of Rambai stem decomposition can be observed from the analysis result:

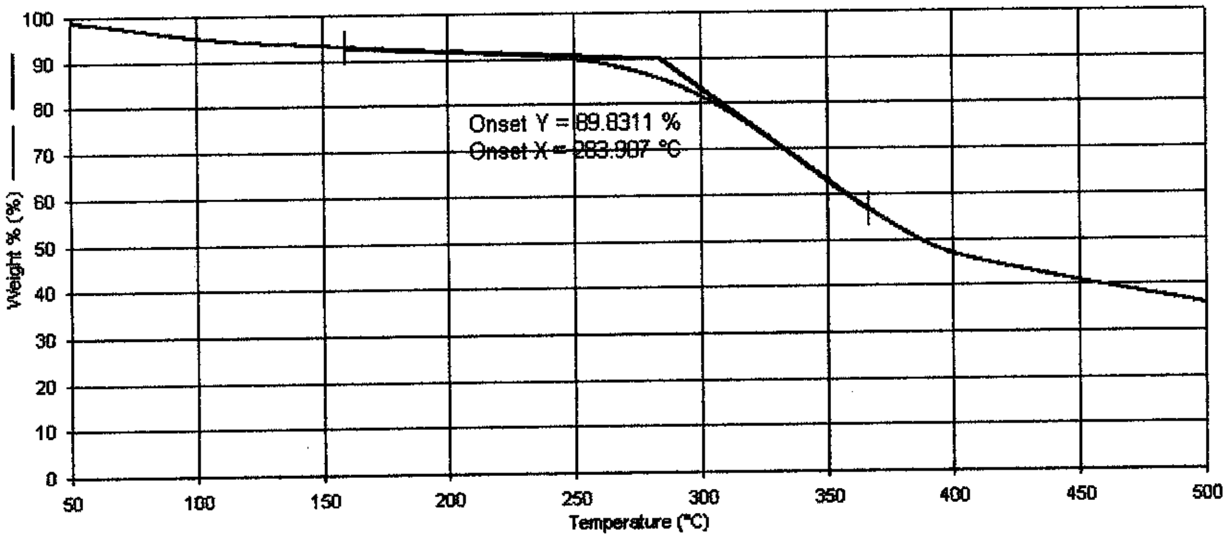
Heating rate: 10 C/min



Heating rate: 15 C/min



Heating rate: 20 C/min



3) Comparison of performance of Rambai stem with other Biomass adsorbent and activated carbon

Table 1. Adsorption of metal ion from synthetic wastewater

Metal solution	Pb^{++}		Ni^{++}		Zn^{++}	
Initial concentration	49.00 mg/L		48.25 mg/L		50.50 mg/L	
Initial pH	5.42		5.46		5.56	
Leaf	**Final conc.**	**Removed%**	**Final conc.**	**Removed%**	**Final conc.**	**Removed%**
Bayberry Wax-Myrtle	7.0	85.7	34.5	28.5	36.0	28.7
Common Persimmon	2.0	95.9	21.5	55.4	18.5	63.4
Common Sassafras	8.0	83.7	35.5	26.4	33.0	34.7
Eastern Redbud	4.0	91.8	29.5	38.9	28.5	43.6
Flowering Dogwood	6.0	87.8	26.0	46.1	18.0	64.4
Glossy Privet	6.0	87.8	31.0	35.8	30.5	39.6
Southern Magnolia	3.5	92.9	35.5	26.4	31.5	37.6
Mountain-Laurel	2.0	95.9	22.0	54.4	20.5	59.4
Sugar Maple	11.0	77.6	18.5	61.7	14.5	71.3
Pecan	10.5	78.6	24.5	49.2	22.0	56.4
Pin Oak	8.0	83.7	33.5	30.6	34.5	31.7
Willow Oak	8.0	83.7	34.5	28.5	35.5	29.7
Activated carbon	3.0	93.9	15.2	68.5	14.1	72.1

*Table obtained from: *Removal of Metal Ions from Wastewater with Natural Wastes*, Adeyinka A. Adeyiga, Liang Hu, Tina Greer School of Engineering and Technology, Hampton University, Hampton.