Adsorptive Removal of Ni(II) Ion from Aqueous Solution Using Rice Husk-Based Activated Carbon

By

Muhamad Shauqie Sulaiman

Dissertation submitted in partial fulfillment of
the requirements for the
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CERTIFICATION OF APPROVAL

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Approved by,

(Mr. Mohd Faisal b. Taha)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH,PERAK

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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.

(MUHAMAD SHAUQIE SULAIMAN)

ABSTRACT

This study is using one of many methods that used to remove heavy metal from industrial. They include chemical precipitation, ion exchange, electroplating, and membrane separation. This study of rice husk as potential activated carbon as a low-cost adsorbent to remove Nickel (Ni) from Ni (II) ion solution was investigated. It used adsorption method as it is cheaper than others and readily available. This project will focuses on the capability of the rice husk as a low-cost adsorbent to remove heavy metal. Raw rice husk, is largely available in Malaysia, as our country main agriculture product is rice. Since rice husk is treated as waste, we are studying of the potential of its' advantages in removing heavy metal. The main components of rice are carbon and silica. Thus it has potential to be converted as an adsorbent. This study involves 3 steps which are the preparation of the raw rice husk, activation stages (treatment of rice husk and carbonization of the treated rice husk), and the kinetic study for the rice husk obtain after activation in order to obtain Langmuir graph. The pores development will be analyzed by using both SEM and FESEM for each stage. Other analyses on rice husk activated carbon were also conducted using BET Surface Area Analyzer, Fourier Transmitted Infrared Spectroscopy (FTIR), CHN Elemental Analyzer, X-Ray Diffraction (XRD) and Thermo gravimetric Analyzer for characterization study on rice husk particle. The adsorption capacity towards metal ion was determined using Atomic Adsorption Spectroscopy (AAS). The result obtained from the adsorption study showed that rice husk has a potential to be converted to adsorbent for removal of heavy metals.

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CHAPTER 1: INTRODUCTION

1.1 PROJECT BACKGROUND

Heavy metals mostly exist in wastewaters which are released from various industries into the environment. It is continuously discharged and creates major global concern. The unnecessary release of heavy metals has posed a great problem worldwide. Common heavy metals such as cadmium (Cd), lead (Pb), cobalt (Co), zinc (Zn) and chromium (Cr) etc. are phytotoxic at both low concentrations as well as very high concentration and are detected in wastewater from mining operations, tanneries, electronics, electroplating, batteries and petrochemical industries as well as textile mill products (Singh et al., 2011) . Since they are non-biodegradable,it can harm human health and ecological systems. The adverse effects caused by these heavy metals are of great environmental concern. These heavy metals are prone to bio-accumulate and end up as permanent additions to the environment. Heavy metals are non biodegradable and accumulate in living organisms thereby causing various diseases and disorders (Bailey et al., 1999). Ni, Cd, Hg, Zn, Pb are some of examples of metals that are produced by industries and pose risk to the resources. Releasing of these metals leads to poor water quality, insufficient supply of drinking water and complicate the water pre-treatment process for drinking water (Mohd F. Taha et al., 2011).

In recent years, various methods for heavy metal removal from wastewater have been extensively studied. This paper reviews the current methods that have been used to treat heavy metal wastewater and evaluates these techniques. These technologies include chemical precipitation, ion -exchange, adsorption, membrane filtration, coagulation–flocculation, flotation and electrochemical methods (Fenglian Fu et al, .2010). All the method except adsorption is not economically feasible for such industries due to its high costs. Adsorption is considered fast, inexpensive and global method thus place interest of developing a low cost adsorbents to be presented in industry.

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. The term sorption encompasses both processes, while desorption is the reverse process. The most common industrial adsorbents are activated carbon, silica gel, and alumina, because they present enormous surface areas per unit weight. Activated carbon is produced by roasting organic material to decompose it to granules of carbon - coconut shell, wood, and bone are common sources. Silica gel is a matrix of hydrated silicon dioxide. Alumina is mined or precipitated aluminum oxide and hydroxide. Although activated carbon is a magnificent material for adsorption, its black color persists and adds a grey tinge if even trace amounts are left after treatment; however filter materials with fine pores remove carbon quite well. Agricultural wastes such as banana pith , mangosteen ,sawdust ,sunflower stalks ,groundnut shells and wheat shell have been used to remove metal ions from water solution (Nasehir Khan et al., 2011) . Plants wastes are aren't expensive as they have low economic value.

As we are in Asia continent, rice is the main food source for the people. Yet, rice husk is considered waste and being disposed to the environment by burn it. Some effort had been made to reduce amount of rice husk without burning it. Recent study shows that rice husk can be used in removing heavy metal. Since studies prove positive results in converting rice husk into activated carbon, a further study is made to study the potential of it to remove heavy metal ions in wastewater for it to exchange current activated carbon which is considered expensive with a low-cost adsorbent.

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

Water is one of the most essential sources in our daily life. It is not only sufficient amount of water is critical, it also deal with quality of the water that being use for food preparation, washing and other daily activities. Today wastewater treatment become one of major profitable company to solve problem regarding polluted water. When dealing as universal solvent and used in industrial processes, it is expected that our water today may contain a diverse range of organic, inorganic, metallic and biological effluents from all classes and properties. Water contaminated with heavy metal ions that exceed allowable limit nowadays being recognize as one of the major and most dangerous pollutant in the environment. This contaminated water maybe come from industrial site for processing such metals example of metal plating and wire manufacturing also come from dissolved metal ions in our daily source water. As toxicity level is increasing, there are some initiatives in finding techniques for remove heavy metals from wastewater. Activated carbon is a good adsorbent as it has high surface area and porous structure, high adsorption capacity and a certain degree of reactivity due to the presence of appropriate functional groups (R. Chand et al,. 2009). Due to the relatively high cost of activated carbon, cost effective adsorbents for treatment of metal contaminated wastewaters are necessary.

Based on recent studies, it is said that activated carbon is one of the possible solution to reduce this problem. Some of the techniques which have been used in the removal of metals from effluents include ion-exchange, chemical precipitation, electro dialysis, electrolytic extraction, reverse osmosis and cementation. These methods are expensive and have the inability to remove metals at low concentration (Bishnoi et al., 2004). Compared to the aforementioned techniques, adsorption has been seen to be a more economically favorable alternative for the removal of metals from aqueous solutions (Zeledon-Toruno et al., 2005). Thus, this research proposed to produce activated carbon from rice husk using adsorption method and loading to get the pattern of its adsorption capacity to reduce and remove heavy metal ions for wastewater treatment.

1.2.2 Significance of the Project

Adsorption is typically used in wastewater treatment to remove toxic or recalcitrant organic pollutants (especially halogenated but also non-halogenated), and to a lesser extent, inorganic contaminants, from the wastewater. Adsorption finds applications in tertiary wastewater treatment as a polishing step before final discharge. Adsorption is commonly used in the treatment of industrial wastewaters containing organic compounds not easily biodegraded during secondary (biological) treatment or toxic. Furthermore, it is being recognized as efficient and economically feasible method to remove heavy metal ions wastewater compared to other methods.

Activated carbon in industry nowadays is considered expensive. Thus, continuous researches had lead to use low-cost adsorbent such as rice husk as an alternative activated carbon. Besides, it is a common plant in agricultural base country like Malaysia. As it is treated as waste and being burned in situ, researchers had made advantage of it to be used as activated carbon. Since the main components of rice husks are carbon and silica, it had the potential to be used as an adsorbent. The inner surface of rice husk is smooth and contains wax and natural fats that provide good shelter for the grain but the presence of these impurities also affects the adsorption properties of rice husk chemically and physically (Chowdhury et al., 2011). Thus, the rice husks need to be treated in order to remove unknown particles that might have reduced the efficiency of the adsorption itself.

1.3 OBJECTIVE AND SCOPE OF STUDY

The scope of this study will be focusing on the adsorption capacity by the activated carbon and its behavior. Below are the objectives of the study;

i. To determine the adsorption capacity of rice husk activated carbon digested with sodium hydroxide (NaOH)
ii. The understand the principles of adsorption
iii. To study the potential of rice husk as an activated carbon for adsorption of Ni(II) ions from aqueous solutions.
iv. To develop Langmuir graph based on the result from the experiment.
v. To get the pore pattern of rice husk throughout the preparation processes.

1.3.1 The Relevancy of the Project

Activated carbon, which is prepared from treated rice husk, had a great potential to be an alternative adsorbent for the removal of heavy metal ions from wastewater. This is very crucial, as it is to ensure that the wastewater meet the requirement proposed by the Department of Environment (DOE) before used it for daily activities, or release it to the open environment. Commercial activated carbon, which used graphite as adsorbent, is limited, and significantly costly, especially to the develop country. Thus, rice husk, which is largely available, cheaper, and suitable for develop country as it is a common plant, can be a great alternative of the commercial activated carbon for removal of heavy metal, Ni(II) ions specifically, by the adsorption process.

1.3.2 Feasibility of the Project within Scope and Time Frame

The goal of this project is to provide a Langmuir Graph with a plotted data of various loading activated carbon in adsorption study. Since the rice husk is readily available, it just need to be cleaned, grinding and sieving to 63μm size before treating with NaOH and carbonized in furnace with Nitrogen flow. All the equipments needed for this project are available in Chemical Department. Furthermore, this project had another 2 lab partners doing same project with different heavy metal. Since all the resources are provided, this project can be considered as a feasible project within the time frame given.

CHAPTER 2: LITERATURE REVIEW

2.1 WASTEWATER & NICKEL ION AS A HEAVY METAL WASTE

Increasing industrialization has been accompanied throughout the world by the extraction and distribution of mineral substances from their natural deposits. Many of these have undergone chemical changes through technical processes and finely dispersed in solutions by effluent, sewage, dump, and dust, into the water, the earth, the air and thus into food chain. In controlling contaminants in the waste water, adsorption or retention mechanisms of pollutants in soils or natural materials have received great attention among researchers. Many natural solids have been studied for use in removal of toxic compounds from water systems or prevention of emission of toxic molecules into the atmosphere (A. Koning, 2007). The control of heavy metals in aqueous systems is an interesting study as these substances are recognized as significant pollutants in soil and groundwater. Industries such as mining operations, tanneries, metal electroplating facilities, ceramic industries, painting operations and battery disposal produce aqueous waste streams contaminated by heavy metals that are discharged into the environment without proper treatment. Among the toxic elements found in the polluted environment are Mn, Fe, Zn, Hg, Cr, Cu, As, Ni, Cd and Pb. These toxic elements are pollutants to soil and groundwater. Heavy metal toxicity can result in reduced mental and central nervous function.

This research is focusing on the removal of Ni(II) ion. Nickel is silvery-white. Hard, malleable, and ductile metal. It is of the iron group and it takes on a high polish. About 65 % of the nickel consumed in the Western World is used to make stainless steel. The total amount of nickel dissolved in the sea has been calculated to be around 8 billion tons. Humans may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel-contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health such as lung cancer, dizziness, respiratory failure, asthma and chronic bronchitis. For animals nickel is an essential foodstuff in small amounts. But nickel is not only favorable as an essential element; it can also be dangerous when the maximum tolerable amounts are exceeded. This can cause various kinds of cancer on different sites within the bodies of animals, mainly of those that live near refineries.

2.2 RICE HUSK AS ADSORBENT

In previous years, there are needs for safe and economical methods for the removal of heavy metals from contaminated waters has necessitated research towards the production of low cost alternatives to commercially available activated carbon. Some agricultural waste products such as rice husk have been shown to have the potential as inexpensive sorbents. The Langmuir adsorption isotherms were used to investigate the adsorption process. The Northeastern region is particularly important as one of the major rice growing belts of the country. The abundance, availability, and low cost of agricultural byproducts make them good adsorbents for the removal of various pollutants from wastewaters. (M. Ahmaruzzaman et al., 2011). By taking case of abundant of agricultural by products, this rice husk can be a good alternative for cheaper adsorbent. Based on about 571 million tonnes rice produced annually which 90 % is produced in developing countries, approximately 140 million tonnes of rice husk available annually for utilization (Mohd F. Taha et al., 2011). This rice husk is not being used by the local, and for the worse case, it will be burned in-situ that may affect the environment. Rice husk,which accounts for 20% by weight of rice, comes from rice milling process as by-product(Thipwimon et al.,2010). Rice husks are rich in organic materials and silica which can be converted to activated carbon. Thus, this make rice husk as a very suitable type of adsorbent.

2.3 ADSORPTION PROCESS

Adsorption process happens in most natural physical, biological and chemical operating system consists two main requirement including adsorbate that is element to be adsorbed and adsorbent the medium for adsorption taking place. Adsorption process involves separation of substance from one phase (adsorbate) and later on will join or accompanied all the surface of another component (adsorbent).

Few related application in industry that apply adsorption process are purification, recovery, and also wastewater treatment. Adsorption at the surface or interface is caused by the binding forces between individual atoms, ions, or molecules of an adsorbate and at the surface, all these forces having their origin in electromagnetic interactions. Adsorption can be apply to chemical and other pollutant recovery base on few types including the best option using activated carbon that can use low material cost and use agriculture waste.

2.4 ADSORPTION ISOTHERMS

In the process of adsorption, adsorbate gets adsorbed on adsorbent. According to Le-Chatelier principle, the direction of equilibrium would shift in that direction where the stress can be relieved. In case of application of excess of pressure to the equilibrium system, the equilibrium will shift in the direction where the number of molecules decreases. Since number of molecules decreases in forward direction, with the increases in pressure, forward direction of equilibrium will be favored. **Figure 1** shows the basic of Adsorption Isotherm.

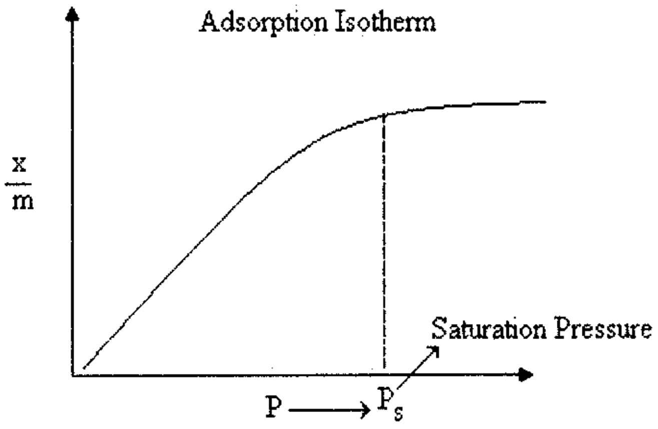

Figure 1: Basic Adsorption Isotherm

From the graph, we can predict that after saturation pressure Ps, adsorption does not occur anymore. This can be explained by the fact that there are limited numbers of vacancies on the surface of the adsorbent. At high pressure a stage is reached when all the sites are occupied and further increase in pressure does not cause any difference in adsorption process. At high pressure, adsorption is independent of pressure.

Figure 2 below shows another adsorption isotherms graph. The concentration in the solid phase is expressed as q, mg g^{-1} solid and in the fluid phase (gas or liquid) as c, mg L^{-1} fluid.

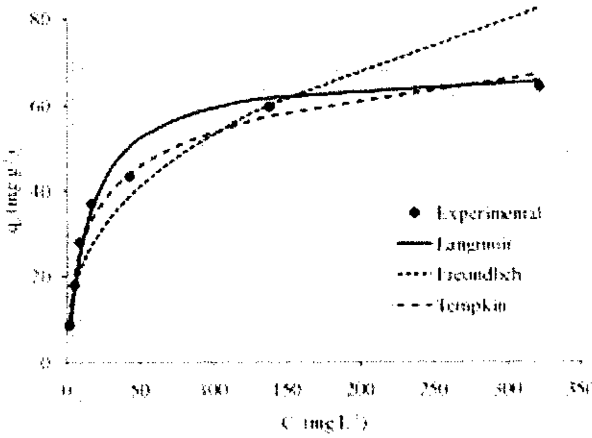

Figure 2: Adsorption isotherms

2.4.1 Langmuir Isotherm

The Langmuir equation which is restricted to Type I isotherms, which represents unimolecular adsorption and applies to microporous adsorbents with small pore sizes. It is derived from simple mass-action kinetics, assuming chemisorptions from either gas or liquid solutions. The Langmuir isotherm model assumes monolayer adsorption on a homogeneous surface. Originally based on a kinetic basis, the model is thermodynamically consistent as it reduces to Henry's law at low pressures (Leszek Czepirski et al, 2000). Assuming that all the adsorption sites are identical and each site accommodates one adsorbate molecule only; the adsorption of adsorbate molecules takes place at well-defined localized states; and that the forces of interaction between absorbed molecules are negligible. The equation for liquid phase adsorption isotherm is as below:

$$q = V(C_o - C) / W \qquad (1)$$

The q symbol represents the adsorbate that adsorbed per unit weight of adsorbent (mg/g), V is the volume of solution (L), C_o is the initial concentration of solution (mg/L) and W is the symbol for the weight of the adsorbent (g). Isotherms are empirical relations, thus it used to predict how much solute can be absorbed by activated carbon. Three well known isotherms are Freundlich, Langmuir, and Linear isotherms. As this research is focusing on water treatment application, the most common used isotherm is Langmuir. Below showed formula of Langmuir isotherm:

$$q = q_o c / (K + c) \qquad (2)$$

where qe is the amount adsorbed per unit mass of adsorbent (wt/wt),qo and K are empirical constants, and Ce is equilibrium concentration of adsorbate in solution after aadsorption. The constants qo and K can be determined by plotting Ce/qe vs. Ce and rewriting equation (2) as:

$$1/q=(K/q_o)(1/c)+1/q_o \qquad (3)$$

2.5 ACTIVATED CARBON

Carbonaceous materials have long been known to provide adsorptive properties. The earliest applications may date back centuries with the discovery that charred materials could be used to remove tastes, colors and odors from water. It is widely known widely used as adsorbents. They are widely used in many applications specifically in removal heavy metal from liquids or gases. Carbon can also be used as a catalyst support for gas-solid reactions, if the temperature and pressure of the gas phase are such that the carbon support is stable under gaseous reactants of the catalytic process(H. Jüntgen.,2003). Activated carbons comprise elementary micro crystallites stacked in random orientation and made by the thermal decomposition of various carbonaceous materials followed by an activation process. The high cost of activated carbon has stimulated interest in examining the feasibility of using cheaper raw materials. Substitute materials tested include straw, automobile tyres, fly ash, coal reject, sewage sludge, bagasse, fertilizer waste and sawdust (S. Rengaraj et al,2002). Activated carbon is also called activated charcoal or activated coal is very porous and has a large surface area that is available for adsorption process.

2.5.1 Classification of Activated Carbon

Generally activated carbon is classified into one of three types, such as powder, granular, and fibrous according to its size and shape, and each type has its specific application.

a) Powdered Activated Carbons

This kind of activated carbon has fine granulometry with average diameter between 15 and 25 μm. Based on their size, obviously they present a large external surface and a small diffusion distance. The rate of adsorption is very high and the problems related to mass transfer are very low. Their low diffusion rate makes them preferably used for adsorption from solution. To minimize the consumption of the carbon, the treatment is occasionally carried out in a countercurrent principle but the process involve large amount of investment.

b) Granulated Activated Carbons

Granulated activated carbons have larger size of carbon particles in the granules compared to carbon powders and so present smaller external surface. Diffusion of the adsorbate plays an essential factor for choosing this kind of adsorbent. These carbons are therefore preferred for adsorption of gasses and vapors base on their good diffusion rate. Adsorption from the gas phase is carried out almost consistently under dynamic conditions by flowing the gaseous mixture and the carrier gas through a bed of a carbon. The size of the carbon granules is also a vital factor for selection where it should not be too small to prevent excessive pressure drop along the bed and carrying away of the carbon particle with the gaseous steam.

2.5.2 Properties of Activated Carbon

The properties of activated carbon are referred to:

i. Its large surface area
ii. A high degree of surface activity
iii. Universal adsorption effect
iv. Favorable pore size as an access route to the internal surfaces
v. Enhanced mechanical strength

The performance of the activated carbon as the adsorbent related in large measure to their intraparticle properties. Surface area and the distribution of area with respect to the pore size generally are the primary determinants of the adsorption capacity. The nature of the intraparticle surface area markedly affectly the types of adsorption interactions that will be operative for an adsorbent, and it is a major distinguishing factor between the activated carbon and the other synthesis adsorbent .The most widely used commercial active carbons have a specific surface area of the order of 600-1200m^2/g. the pore volume limits the size of the molecules that can be adsorbed whilst the surface area limits the amount of material which can be adsorbed, assuming a suitable molecular size. The adsorptive capacity of adsorbent is related to its internal surface area and pore volume. If the surface area is high, the adsorptive capacity will be high. These provide a large surface area relative to the size of actual carbon particle and its visible exterior surface.

The structure of an activated carbon is composed of pores classified into three groups, namely micro pores, mesopores and macropores. Micropores usually account for over 95% of the total surface area of activated carbons. The volumes of the micropores range from 0.15 up to 0.6 cm^{3}· Conventional activated carbons are tridisperse,having all three types of pores present within their structure (Nevin Yalçın et al, 2000).

According to IUPAC:

- Micropores : below 1 nm radius
- Mesopores : 1-25 nm radius
- Macropores : radius > 25 nm

At most time, adsorption only occurs when in micropores. The macropores will determine the accessibility of the adsorbent, and the mesopores will help in transport of the adsorbate from the gas phase to the micropores. An adsorbent with a high activation degree, and therefore a high total pore volume, will possess a high maximum adsorption capacity.

2.5.3 Chemical Structures of Activated Carbon

Carbon materials are activated by a series of processes which include:

i. Removal of all water (Dehydration)

ii. Conversion of the organic matter to elemental carbon, driving-off the non carbon portion (carbonization)

iii. Burning off tars and pore enlargement (activation)

Activated carbon's structural unit is depends on the structure of pure graphite. The weak van de Waals forces held layer of fused hexagons that composed the graphite crystal. The layers are held by carbon-carbon bonds. Activation carbon is a disorganized form of graphite, due to impurities and the method of preparation (activation process).

2.6 FACTORS AFFECTING ADSORPTION EFFICIENCY

The ultimate goal for every research related to activated carbon is to know the adsorption capacity that determined the effectiveness of such activated carbon. Factors that listed below had been considered that impact on experiment result.

2.6.1 Characteristic of adsorbate

The chemical composition of the adsorbate is important. This character determines the size and the composition of molecule that want to be adsorbed as well as the adsorption efficiency. Molecular size is important as the solubility will decreases as the size is increases. Molecular size is also important as the adsorbents depend on internal surface area for full use of the adsorption capabilities. If the size is too large, the adsorption capacity will decrease as large molecules cannot penetrate pores within the adsorbent. Another aspect is that the larger molecules will diffuse slowly from solution and will results in longer time for full equilibrium adsorption capacity. This leads to the conclusion that the amount adsorbed increases with decreasing particle size and higher surface area (V.K.Gupta et al,2011)

2.6.2 pH of Activation Agent

The pH of a solution is significance for its effect on the adsorbent, as well as on the adsorbate. Some adsorbents may have affinity for H+ and OH- ions and can directly affect the solution pH, therefore will affect solubility and adsorption capacity. This effect must be considered during test design where large quantities of adsorbent may be used per volume of solution. The pH change may not be so efficient in full-scale operation where large volumes of solution are treated and not giving so many impacts. Study from K.Y.Foo and B.H. Hameed shows that it was found that increasing solution pH serves to increase the adsorption capacity, with a significant enhancement as the pH increased from 6 to 8.

2.6.3 Carbonization Time of Rice Husk

The adsorption activity is reported to be decreased as the carbonization time increases, due to the thermal destruction of pores structure as carbonization time increases. An increased in the burning time increased the amount of silica but decreased the amount of carbon content will predictably cause the decrease of the porosity of the adsorbent. This is expected since at longer times, more carbon are converted to silica (Daffala, Mukhtar & Shaharun,2010)

2.6.4 Carbonization Temperature of Rice Husk

Based on previous study, the purpose of carbonizing the rice husk is to ensure that there is thermal decomposition. From there, non-carbon species will be eliminated and carbon mass will be fixed. Chemical activation is usually carried out at temperature between 400 and 800°C. There is an optimum temperature which is different for different types of raw materials. Some studied is needed in order to find the optimum activation temperature for a particular raw material. The previous study was varying the temperature and the results shows that at 500°C, it is optimized.

2.6.5 Contact Time of Adsorption Activities

The contact time will affect the heavy metal adsorption on rice husk activated carbon. As the contact time increases, the metal ion adsorbed will also increase. The oil adsorption was fast at the initial stages of the contact period, and thereafter it become slower near the equilibrium. This phenomenon was due to the fact that a large number of vacant surface sites were available for adsorption during the initial stage, and after a lapse of time, the remaining vacant surface sites were difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases (H.H. Sokker et al,2011)

2.6.6 Effect of Agitation

The adsorption experiments were carried out as function of time, initial concentration and agitation speed. The equilibrium data fits well to Langmuir adsorption isotherm. This is due to the increased turbulence and as a consequence, the decrease boundary layer thickness around the adsorbent particles as a result of increasing the degree of mixing. At 300rpm the adsorption capacity was maximum and beyond this there was no significant increase in adsorption capacity. Based on study made by Hassina Zaghouane-Boudiaf et al (2011), it shows that the adsorption increases with an increase in stirring time and attains equilibrium.

CHAPTER 3: METHODOLOGY

3.1 PROCEDURES

The whole process or methodology of the study can be separated into four vital parts:

i. Preparation of raw rice husk
ii. Treatment of raw rice husk
iii. Carbonization of treated rice husk
iv. Adsorption study

Based on the first three parts, they are the methods that are involved in preparing rice husk based activated carbon. The final part of the experiment is performing adsorption study with Nickel Chloride, NiCl as the adsorbate. The adsorption capacity activity will be observed, as to prove that rice husk can be a potential adsorbent. About three kilograms of raw rice husk were obtained from Perak to be used as feedstock of this study.

3.1.1 Preparation of Raw Rice Husk

Before the process of obtain the desired size of rice husk, cleaning and drying are done.

The brief procedures of this process are stated below:

i. About 500 g of raw rice husk was washed with distilled water several times to remove any adhering materials.
ii. Rice husk was dried in the oven at 100°C for 24 hours.
iii. The dried rice husk was grinded using mortar grinder
iv. Grinded rice husk was then sieved using a set of sieve trays sized 63 μm to obtained the exact size of 63 μm.

3.1.2 Treatment of Raw Rice Husk

1.0 M of Sodium Hydroxide (NaOH) was used as activation agent for the rice husk treatment process. The required NaOH was prepared by dissolving 40 g of NaOH pellets in 1 L of distilled water. The ratio of rice husk to the digestion agent was 1:10 where rice husk is in unit of gram (g) while digestion agent is in unit of milliliter (ml).

The procedures for treatment of rice husk are as follows:

i. 1.0 M NaOH was prepared.
ii. 50 g of raw rice husk size 63 μm was digested with 500 ml of 1.0 M NaOH in 600 ml beaker.
iii. Beaker containing sample was sealed using aluminium foil.
iv. After 24 hours, the digested sample was filtered and washed with excessive deonized water until the filtrate is free from base (value of pH will be around 6 to 7)
v. The sample was placed on the tray for the drying process in an oven at 100°C for 24 hours.
vi. After the drying, the sample was weighed.

*Preparation of NaOH is to be conducted in fume hood for safety purpose.

3.1.3 Carbonization of Treated Rice Husk

The treated rice husk are carbonized to impact thermal decomposition to it thus eliminating non-carbon species and fixed the carbon mass as well as the basic carbon structure. A total of about 40 grams of treated rice husk are carbonized.

The steps of carbonization are as follows:

i. About 40 g of treated rice husk was weighed.
ii. Aluminium foil was used to wrap the treated rice husk.
iii. Wrapped sample was placed into the crucible and crucible lid was used to close crucible to minimize the effect of oxidation.
iv. The crucible that contain rice husk was put into the furnace when the temperature reaches nearly around 500°C.
v. After carbonizing the rice husk for 2 hours, furnace temperature was reduced to 25°C.
vi. Samples were taken out once the furnace has reached the desired temperature.
vii. After that, the samples were weighed and the percentage of loss of weight was calculated.

3.1.4 Adsorption & Extraction Study

The purpose of the adsorption process is to know the adsorption activities. This study was carried out by mixing 0.1 g of sample in 25 ml of 150 ppm metal ion solution. The contact time was ranging from 30 to 150 minutes. From that, the sample was then filtered to get the filtrate before analyzed using Atomic Adsorption Spectrophotometer (AAS).

These are the procedures that will be conducted to study the adsorption capacity of rice husk based activated carbon using AAS.

I. 25 ml of 150 ppm Ni^{2+} was prepared and mixed with 0.1g of carbonized rice husk in the conical flask.
II. Conical flask was placed in waterbath shaker and left for stirring at 170 rpm, 25°C for 5 minutes.
III. The solutions were then filtered using vacuum filter and the filtrate is analyzed using AAS to check the metal concentration left in the nitrate.
IV. Step 1 to 3 was repeated for various contact time ranging from 5, 10, 15,20,30,60,90 and 120 minutes.
V. Graphs were plotted to observe the effect of parameters varied on the adsorption activities

3.1.5 Analyses

Samples before and after extraction study are analyzed by using Scanning Electron Microscope (SEM), Atomic Absorption Spectrophotometer (AAS), Fourier Transform Infrared (FTIR), Thermogravimetric Analyzer(TGA), CHN Elemental Analyzer, and X-Ray Diffraction (XRD). Below is the list of samples (for reference) which were sent for analysis.

Table 1: List of samples for analysis study

No.	Sample Name & Description	Label
1	Raw Rice Husk	RRH
2	Treated Rice Husk	TRH
3	Carbonized Rice Husk at 500°C	CRH

Scanning Electron Microscope (SEM)

The SEM is a microscope that uses electrons rather than light to form an image. It produces images of high resolution, which means that closely spaced featured can be examined at high magnification. Preparation of the sample is relatively easy since SEM only required the sample to be conductive. The development of pores can be easily observed due to the combination of higher magnification, larger depth of focus and great resolution.

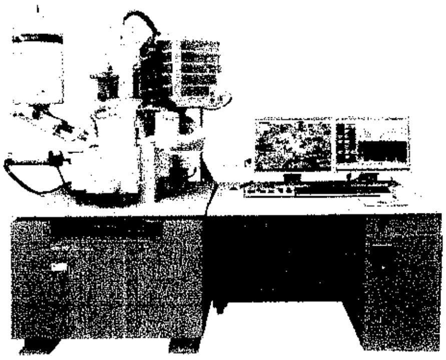

Figure 3: SEM

Atomic Absoprtion Spectrophotometer (AAS)

AAS is and analytical equipment that used for research works. It give definite reading of metal ion concentration. AAS functioning by giving accurate quantitative analyses for metals in water,sediment soils or rock. In this study, we use AAS to determine final concentration on metal ion in filtrate.

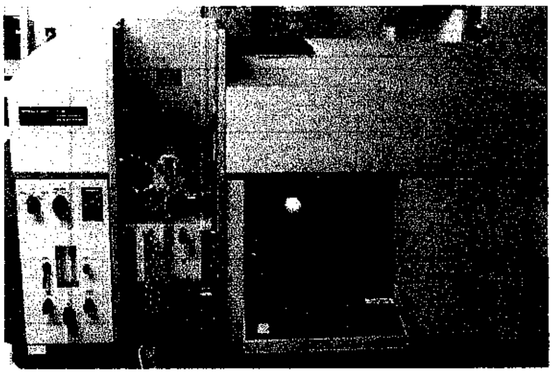

Figure 4: AAS

CHN Elemental Analyzers

CHN stands for carbon,hydrogen,and nitrogen. This analyzers provide means for rapid determination of carbon,hydrogen and nitrofen in organic matrices and other types of materials. It is functioning to give carbonate and organic carbon and get some idea of the composition of organic matter.

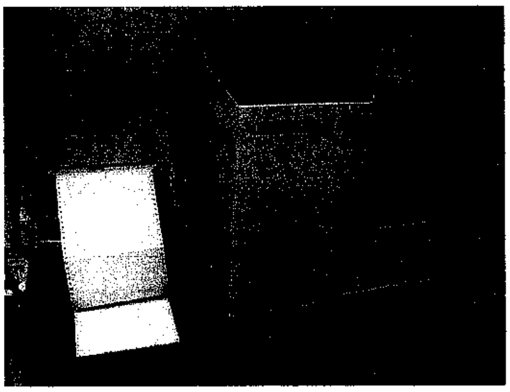

Figure 5: CHN Analyzers

Fourier Transform Infrared (FTIR)

FTIR is the preferred method of infrared spectroscopy. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. The size of the peaks in the spectrum is a direct indication for amount of material that presents in the sample. The main purpose of performing FTIR analysis here is to identify functional group of the samples.

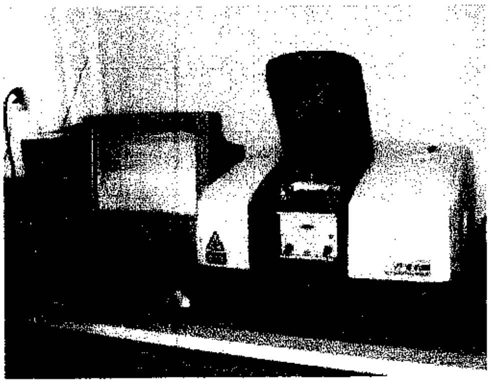

Figure 6: FTIR

Thermogravimetric Analyzer (TGA)

TGA measure the amount and rate of change in the weight of a material as a function of temperature or time in a controlled temperature. The measurements are used primarily to determine the composition of materials and to know the thermal stability of temperature at 1000°C. TGA also can characterize the material that exhibit weight loss or gain due to composition, oxidation or dehydration.

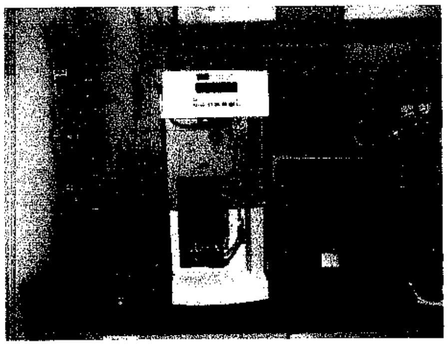

Figure 7: TGA

X-ray Diffraction (XRD)

The principal function of XRD is to know the diffraction beam intensity and the d-spacing's of crystalline materials, which however had been developed to determine many structural properties. In this study, XRD is conducted to investigate the crystalline and amorphous phase of the carbon samples. XRD experiments are performed with 2Θ values ranging from 2° to 80°.

Figure 8: XRD

3.2 EQUIPMENTS AND CHEMICALS

Lab equipments needed in this project are as follows:

Table 2 : List of Equipments

No.	Equipment	Quantity	Remarks
1	AAS	As per requirement	
2	SEM	As per requirement	
3	CHN	As per requirement	
4	FTIR	As per requirement	
5	TGA	As per requirement	
6	XRD	As per requirement	
7	Electrical Siever	As per requirement	
8	Grinder	As per requirement	
9	Oven	As per requirement	
10	Furnace	As per requirement	with Nitrogen Flow
11	pH meter	As per requirement	
12	Waterbath Shaker	As per requirement	
13	Blender	As per requirement	

The chemicals involved in this project are listed below:

Table 3: List of Chemicals required

No.	Chemical	Quantity	Remarks
1.	Sodium Hydroxide,NaOH	As per requirements	
2.	Nickel Chloride,$Ni(Cl)_2$	As per requirements	Adsorbate
3.	Distilled water	As per requirements	

3.3 PROJECT ACTIVITIES

Table 4 : Project activities planned for Final Year Project

Activities	**Starting Month**	**Finishing Month**
Survey on the availability of suggested Experiment Apparatus	1st Apr 2012	4th Apr 2012
Study on method to remove heavy metal ions by using rice husk	5th Apr 2012	14th Apr 2012
Study on adsorption method that remove Nickel ion using rice husk	15th Apr 2012	28th Apr 2012
Getting the source of rice husk availability	29th Apr 2012	11st May 2012
More study on the method	12nd May 2012	21st May 2012
Experiment preparation	22nd May 2012	31st May 2012
Experiment on sieving rice husk to 63μm	1st Jun 2012	15th Jun 2012
Experiment on treated rice husk	16th Jun 2012	31st Jun 2012
Experiment on carbonization of rice husk	16th Jun 2012	31st Jun 2012
Analysis of the data/adsorption study/kinetic study	1st Jul 2012	30th Jul 2012
Report documentation	3rd Aug 2012	30th Aug 2012

3.4 GANTT CHART & KEY MILESTONE

Table 5 : Gantt Chart & Key Milestone

	FYP 1				FYP 2			
Activity	J	F	A	M	J	J	A	S
Selection of Project Topic	■	■						
Preliminary Research Work		■						
Submission of Extended Proposal Defense			■					
Survey on the availability of suggested Experiment Apparatus			■					
Purchase unavailability things. Study on how to prepare the solutions.			■					
Defense proposal. Present details on methodology of the experiment.			■					
Experiment preparation				■				
Submission of Interim Draft Report				■				
Submission of Interim Report				■				
Sieving rice husk to 63 µm					■			
Experiment on treated rice husk					■			
Experiment on carbonization of rice husk					■			
Adsorption study						■		
Analysis of the data/ Kinetic study						■		
Report documentation							■	■

CHAPTER 4: RESULTS AND DISCUSSIONS

This Chapter shows the finding and discussion from the result obtained during the lab work of rice husk activated carbon (RHAC) preparation and the adsorption study of Ni (II) ions using RHAC.

4.1 PREPARATION OF RAW RICE HUSK

As the preparation start, rice husk was cleaned with distilled water. Based on the observation, a lot of impurities that floats on water surface will be removed while the rice husk were stay at the bottom. The main objective of cleaning process is to get rid of any foreign substances that may harm and contaminate the rice husk itself. Then, after the grinding and sieving, amount of 63 μm rice husk that was collected is about 180.30 g. the color of 63μm raw rice husk was dull brown

4.2 TREATMENT OF RAW RICE HUSK WITH SODIUM HYDROXIDE

Using ratio 1:10, 180 g of raw rice husk is mixed with approximately 1800 ml NaOH. This treatment of rice husk was done for 14 hours. Observation on no color change and mass of the rice husk was recorded.

Table 6: Observation results of treated rice husk

	Color	Weight (g)
Before	Dull brown	180.00 g
After treatment	Dull brown	93.42 g
Weight loss (after treatment-before)		86.58 g

Final pH for treated rice husk after washing with excessive distilled water was 7.34.

4.3 CARBONIZATION OF TREATED RICE HUSK

The carbonization process was conducted at 500°C with nitrogen flow of 0.22 m^3/s as inert gas. About 86.58 gram of treated rice husk was carbonized at the desired temperature. The color of the rice husk after the carbonization is changed to dark black. The weight of the sample decrease a lot as volatile matter lost during the carbonization. The results are shown in tables 7 and 8

A summary of carbonization result is shown below.

Table 7: observation result of carbonized rice husk

Temperature (°C)	Color		Weight (g)	
	Before Carbonization	After Carbonization	Before Carbonization	After Carbonization
500	Dull brown	Dark Black	86.58	14.21 g

Table 8: observation result of carbonized rice husk percentage weight loss

Temperature (°C)	Color		Percentage Weight Loss (%)
	Before Carbonization	After Carbonization	
500	Dull brown	Dark Black	83.59

Weight of samples after carbonization was reduced due to elimination of certain elements such as nitrogen and oxygen in the rice husk during carbonization process. Removal of volatile component is necessary to produce a better activated carbon.

4.4 ANALYSES

4.4.1 Field Emission Scanning Electron Microscope (FESEM)

Three samples of rice husk were analyzed using FESEM which are raw rice husk, rice husk treated with 1.0 M NaOH and carbonized rice husk at 500°C.

Figure 9: Raw rice husk (1000 times magnification)

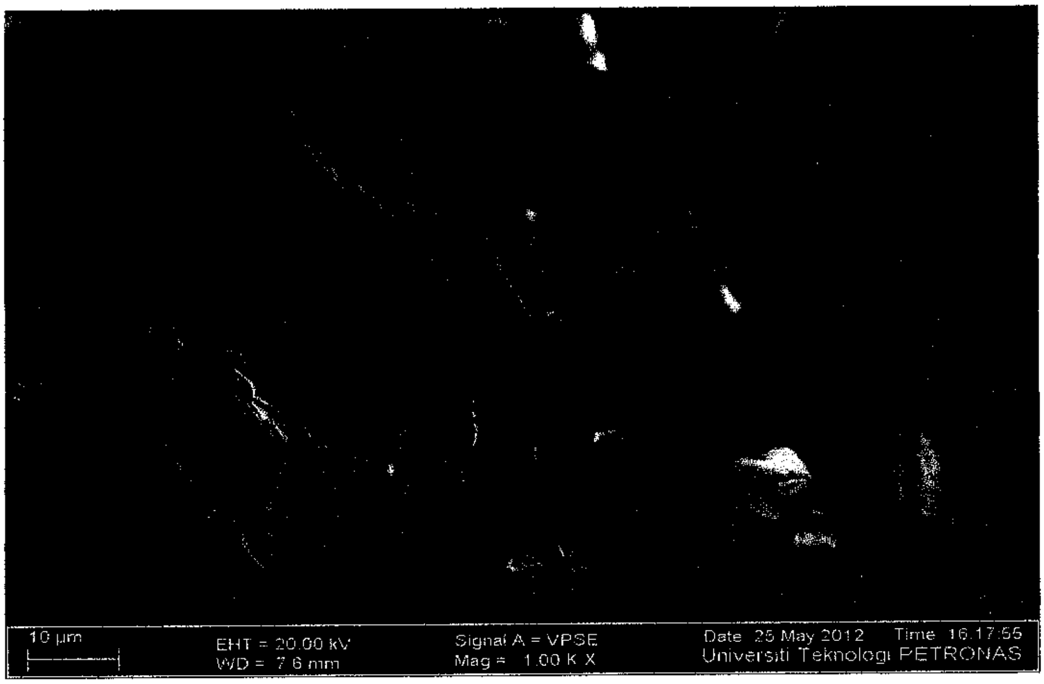

Figure 10: Raw rice husk treated with 1.0 M NaOH (1000 times magnification)

Figure 11: Carbonized rice husk at 500°C (1000 times magnification)

The FESEM micrographs of raw rice husk, treated rice husk and carbonized rice husk are shown in figure 9-11. The samples were different between three of them, thus different morphology was obtained. The pores of rice husk were developed throughout processes, i.e treatment and carbonization process. Figure 9 shows no pores at all while obvious pores can be seen on the treated rice husk as shown in figure 10. Treatment becomes the starting point of the pore development.

4.4.2 Energy-Dispersive X-Ray Spectroscopy (EDX)

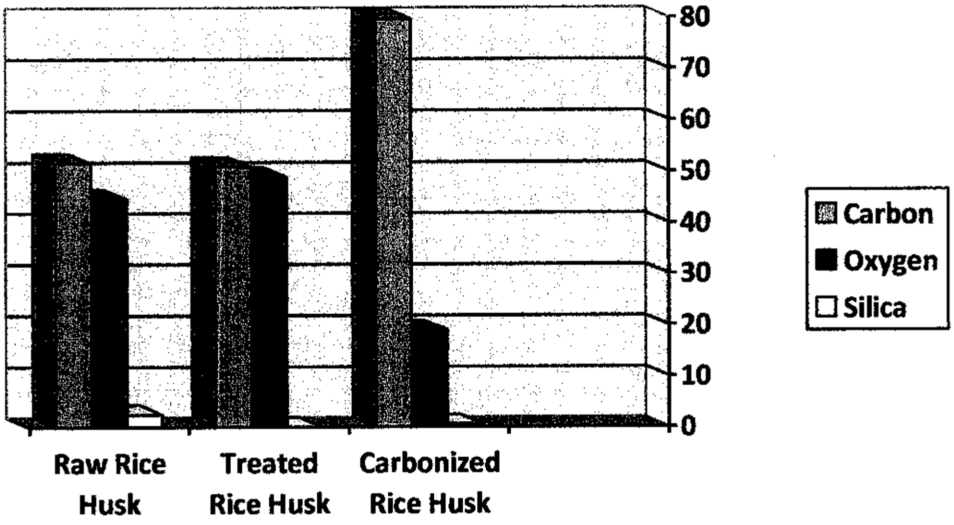

Figure 12: Graph of EDX analysis result

Figure 12 shows change in the compositions of the three samples that been analyzed. Generally, treatment of rice husk with NaOH and carbonization at temperature of 500°C will increased in amount of carbon and reduced amount of oxygen. The silica content reduced till 0.00 after treatment with 1.0M NaOH as compared to raw rice husk as it is being washed with distilled water. Reaction of silica with NaOH forming sodium silicate can be removed by washing with water due to its solubility in water. However, there was slight increment in silica content after the carbonization process.

4.4.3 Fourier Transform Infrared (FTIR)

Figure 13: FTIR Spectra of samples: Raw rice husk

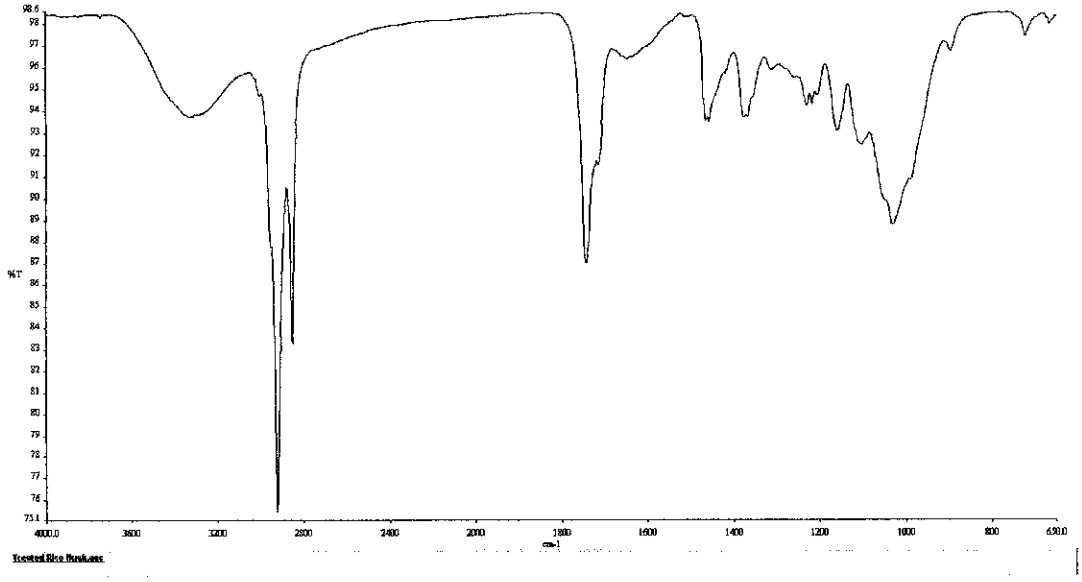

Figure 14: FTIR Spectra of samples: Treated rice husk

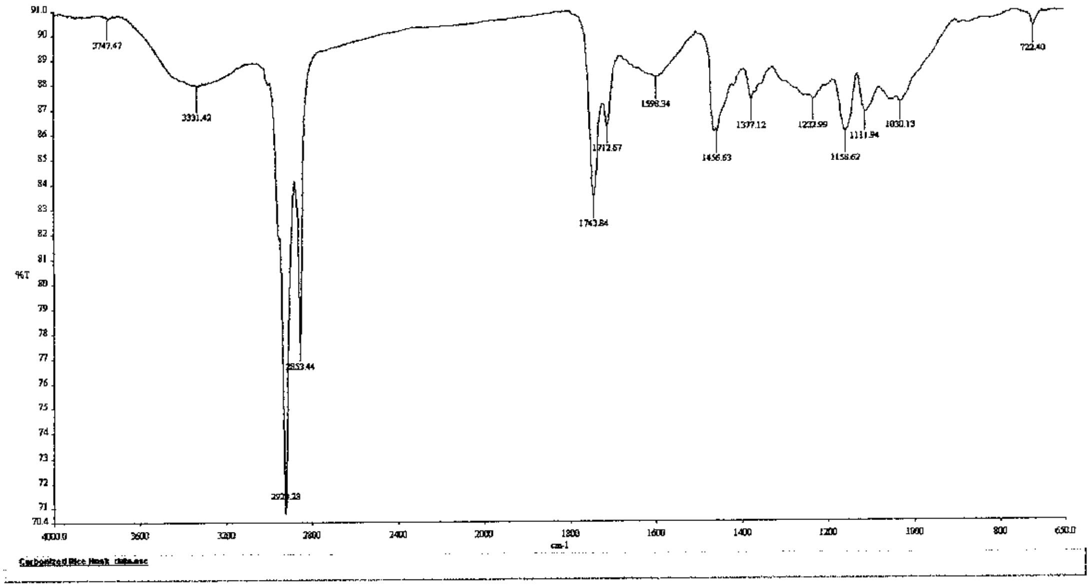

Figure 15: FTIR Spectra of samples: Carbonized rice husk

The FTIR technique is very important to identify the characteristic functional groups. FTIR provides information on the functional groups of rice husk samples and commercial activated carbon (powder) analyzed. By referring to Figure 13, RRH samples show a wide band at about 1300-1000 cm$^{-1.}$ The free C-O & C-H bend monosubstituted stretching mode of hexagonal groups can be assigned to that band. The position and asymmetry if the band at the lower wave numbers indicates the presence of strong Oxygen bonds.

At figure 14, there is a huge band at 3200-2800 cm^{-1} and a loss of band of C-H bond at 1300-1000 cm$^{-1.}$ This indicates the increasing amount of alkanes group, which is C-H. The main reason is that, the rice husk is being treated by NaOH that reduce amount of Silica in the rice husk. The C-H bonds increase as the amount of carbon in rice husk increased after treating process. From 1800-1000 cm^{-1}, there are so many small peaks that exist after the treating process. This happens as the treating agent, NaOH react with all the elements in the rice husk and produce impurities.

As for figure 15, the huge band of 3200-2800 cm^{-1} keeps increasing as C-H bond increase. This is because the carbonization processes increases the amount of carbon for the rice husk and reduce the other elements. The other elements are reduced drastically as organic matter is vaporizing. The changes showed evidence of the formation of structures containing multiple carbon-carbon bonds as well as the elimination of originally present oxygen and hydrogen atoms (Chen et al., 2011). As a conclusion, carbonization helps to increase amount of carbon content.

4.4.4 CHN Elemental Analyzer

Three samples were analyzed using CHN Elemental Analyzer which are raw rice husk, treated rice husk with 1.0 M Sodium Hydroxide, and carbonized rice husk at 500°C for 2 hours. Results of CHN analysis is presented in Table 9.

Table 9: Table of CHN analysis result

Sample	**Carbon %**	**H_2 %**	**N_2 %**	**S %**
RRH	32.24	4.1815	1.538	0.0845
TRH	39.25	4.373	0.7005	0.0000
CRH	65.415	3.8605	1.08	0.0375

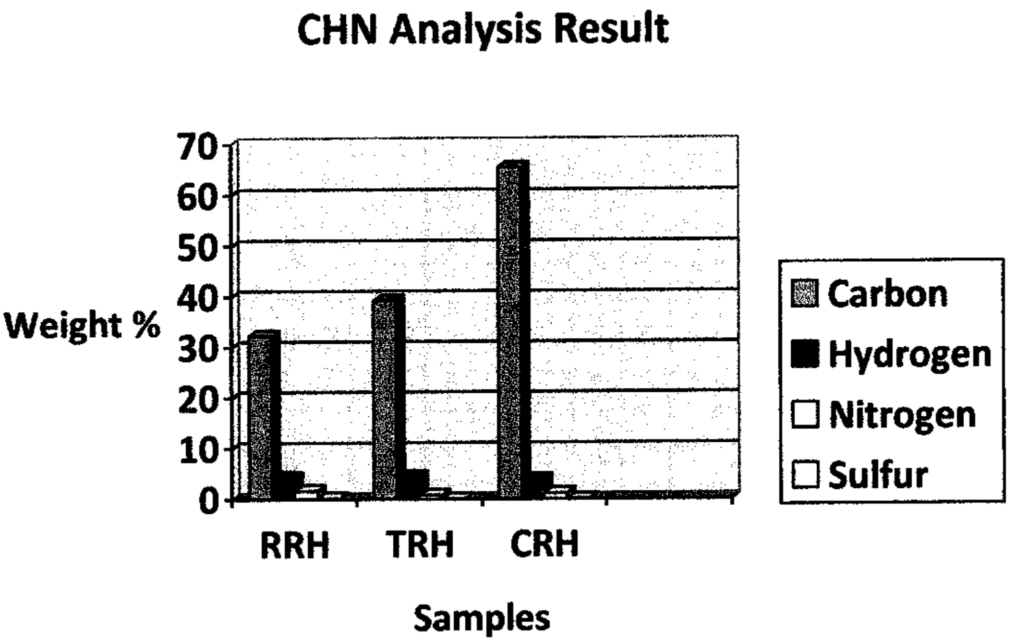

Figure 16: Graph of CHN analysis result

Based on the result obtained, it can be observed that the percentage of the carbon element had increased after the treatment with 1.0 M NaOH and carbonization at 500°C. as the carbon amount increased, we can see a different trend in percentage of hydrogen and nitrogen that is reduce. The reduction of percentage of Hydrogen and nitrogen increase the weight percent of carbon.

4.4.5 X-Ray Diffraction (XRD)

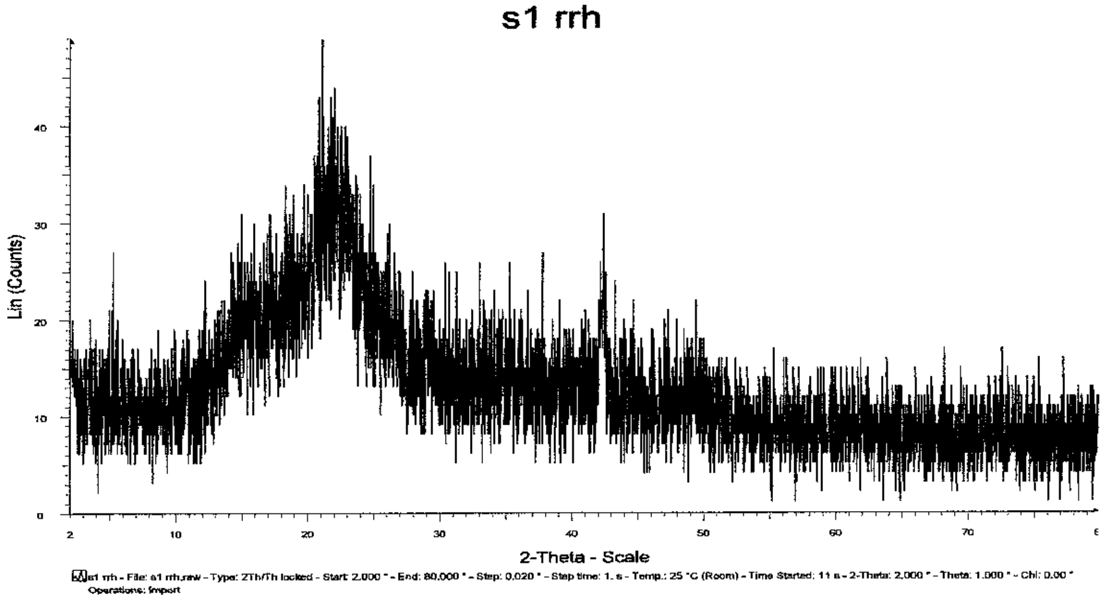

Figure 17: XRD curve of Raw Rice Husk (RRH)

Figure 17 shows the XRD curve for raw rice husk (RRH) while figure 18 and 19 shows the XRD curve for treated rice husk (TRH) and carbonized rice husk (CRH). Based on the results obtained for both RRH and TRH, it can be said that both shows different curves. As for XRD analysis, the crystallinity part will gave sharp narrow diffraction peak and the amorphous component gave a very broad peak (halo). RRH sample indicate the presence of completely amorphous silica by the appearance of a broad peak centered at the 2θ angle of 23°. The broad and sharp peak between degree 2θ 14° and 26° in XRD curve shows that it has a mixture of amorphous and crystalline of silica. The crystalline phases of silica are identified as crytobalite and tridymite (Chen et al., 2011)

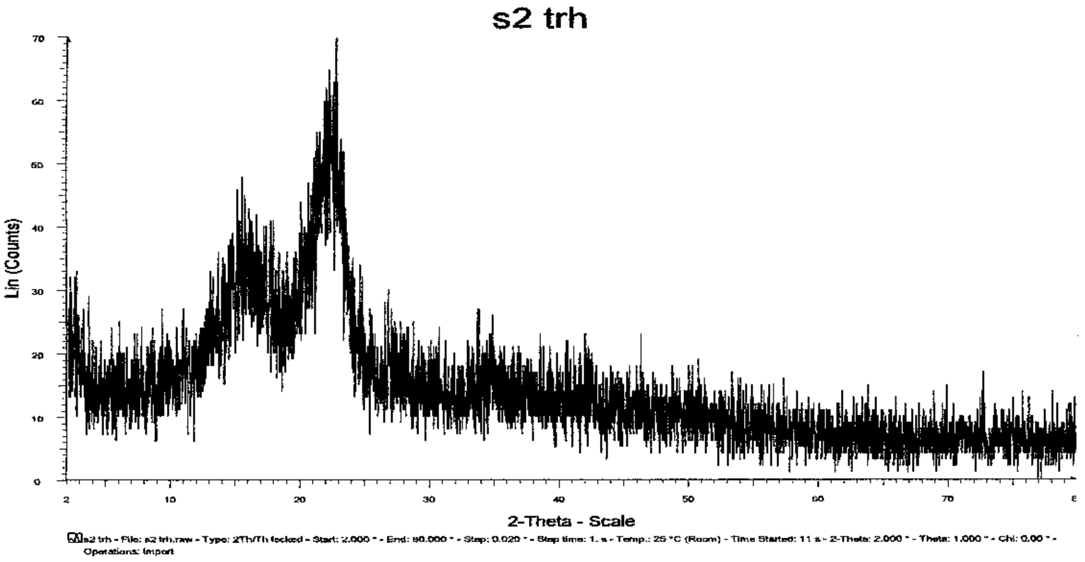

Figure 18: XRD curve of Treated Rice Husk (TRH)

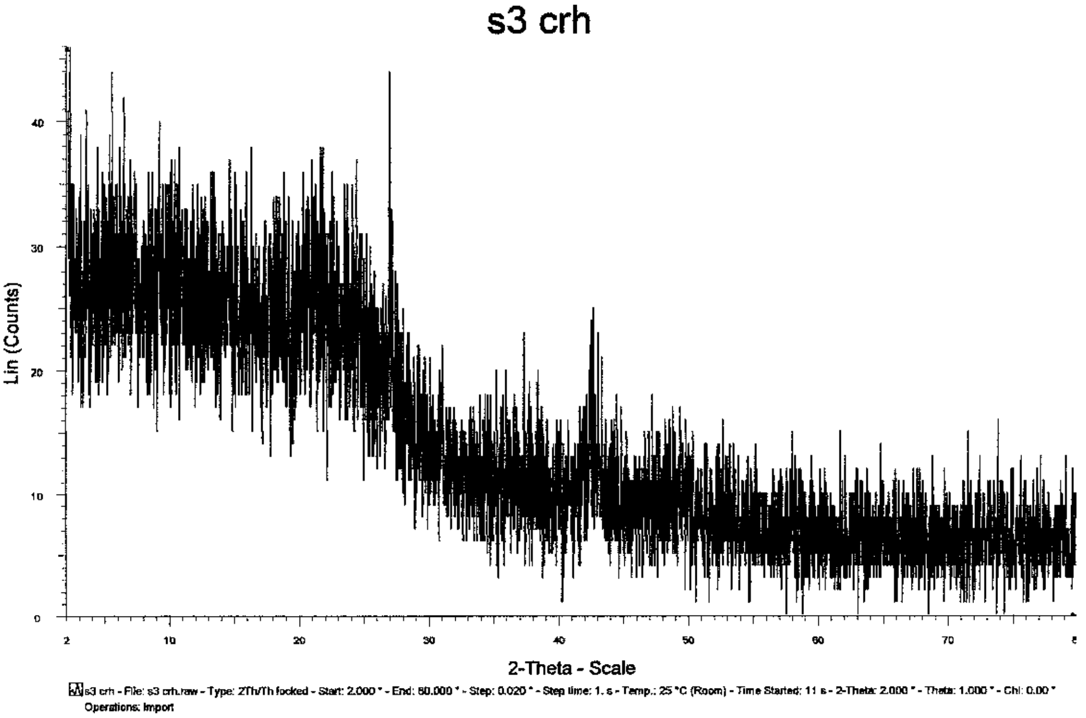

Figure 19: XRD curve of Carbonized Rice Husk (CRH)

4.4.6 Atomic Absorption Spectrophotometer (AAS)

The solution of Ni (II) ions turned from light green to black when rice husk activated carbon (RHAC) were put into the solution shaking process of water bath shaker. After filtering the solution, filtrate solution was analyzed using AAS to check the final concentration of Ni(II) ion. Standard solution of Ni(II) ions with different concentrations of 2 ppm, 4 ppm, 6 ppm, and 8 ppm were prepared in order to obtain the calibration curve. Figure below shows the calibration curve of standard solution

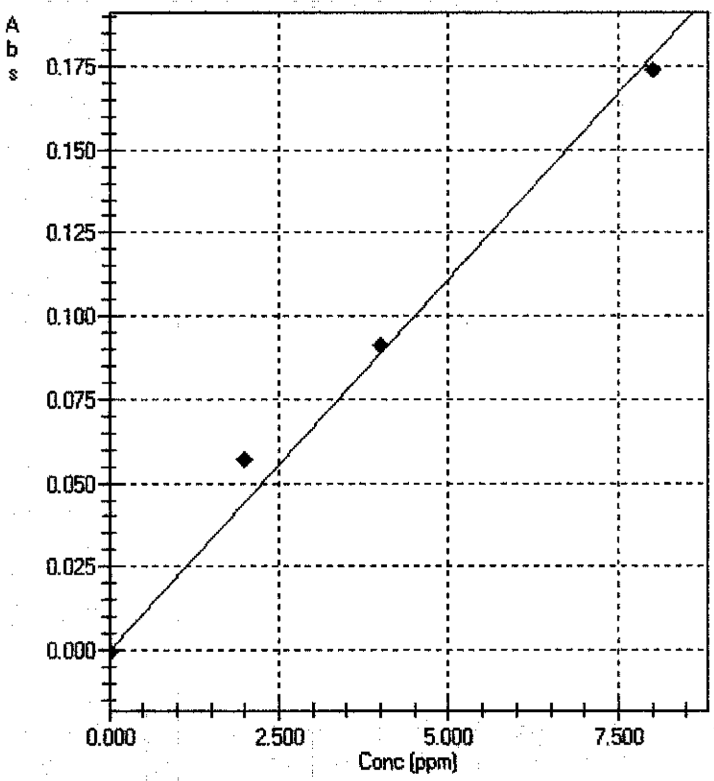

Figure 20: AAS calibration curve with R=0.9963

From the graph shown above, the value of R indicates the deviation of the data collected compared to the straight line (linear). The value of R is obtained from the graph was 0.9963 and indicated that the solution prepared was considerably good as its value was close to 1.

4.5 ADSORPTION STUDY

The results of adsorption study was obtained from AAS are presented in this section. The adsorption study was conducted at varied contact time (5,10,15,20,30,60,90,120 minutes). Contact time mentioned here is referred to the duration of adsorbent that mixed and left in the solution adsorbate. In this study, adsorbent was activated carbon and Ni(II) as the adsorbate. The contact time is the time duration of 0.1 g, 0.2 g, 0.3 g, 0.4 g, and 0.5 g of RHAC mixed with 25 ml of 150 ppm of Ni(II) ion solution left in the waterbath shaker.

Table 10 : Experimental data for the effect of contact time (0.1 g)

Adsorption Contact Time (min)	**Initial Concentration, Co(ppm)**	**Final concentration, Ce(ppm)**	**Percent Metal Uptake (% removal)**	**Adsorptive Capacity, (mg Ni(II) ion adsorbed/g adsorbent)**
5	150	60.3091	59.79	22.42
10	150	61.0317	59.31	22.24
15	150	60.2777	59.81	22.43
20	150	60.5111	59.66	22.39
30	150	60.4258	59.72	22.39
60	150	60.8342	59.44	22.29
90	150	60.5066	59.66	22.37
120	150	61.337	59.10	22.17

Table 11 : Experimental data for the effect of contact time (0.2 g)

Adsorption Contact Time (min)	**Initial Concentration, Co(ppm)**	**Final concentration, Ce(ppm)**	**Percent Metal Uptake (% removal)**	**Adsorptive Capacity, (mg Ni(II) ion adsorbed/g adsorbent)**
5	150	59.7704	60.15	11.28
10	150	59.5281	60.31	11.31
15	150	59.7076	60.19	11.29
20	150	59.4742	60.34	11.32
30	150	59.5325	60.31	11.31
60	150	59.5415	60.30	11.31
90	150	59.4832	60.34	11.31
120	150	59.2587	60.49	11.34

Table 12 : Experimental data for the effect of contact time (0.3 g)

Adsorption Contact Time (min)	**Initial Concentration, Co(ppm)**	**Final concentration, Ce(ppm)**	**Percent Metal Uptake (% removal)**	**Adsorptive Capacity, (mg Ni(II) ion adsorbed/g adsorbent)**
5	150	59.2498	60.50	7.56
10	150	58.976	60.68	7.59
15	150	59.1645	60.56	7.57
20	150	59.0523	60.63	7.58

30	150	59.2498	60.50	7.56
60	150	59.0702	60.61	7.58
90	150	58.6573	60.90	7.61
120	150	59.0612	60.63	7.58

Table 13: Experimental data for the effect of contact time (0.4 g)

Adsorption Contact Time (min)	**Initial Concentration, Co(ppm)**	**Final concentration, Ce(ppm)**	**Percent Metal Uptake (% removal)**	**Adsorptive Capacity, (mg Ni(II) ion adsorbed/g adsorbent)**
5	150	58.9224	60.72	5.70
10	150	58.9501	60.70	5.69
15	150	58.7210	60.85	5.70
20	150	58.8367	60.76	5.70
30	150	58.6473	60.90	5.71
60	150	58.7397	60.84	5.71
90	150	58.6533	60.90	5.71
120	150	58.5965	60.94	5.71

Table 14 : Experimental data for the effect of contact time (0.5 g)

Adsorption Contact Time (min)	**Initial Concentration, Co(ppm)**	**Final concentration, Ce(ppm)**	**Percent Metal Uptake (% removal)**	**Adsorptive Capacity, (mg Ni(II) ion adsorbed/g adsorbent)**
5	150	58.2013	61.20	4.59
10	150	57.9974	61.33	4.60
15	150	58.1348	61.28	4.59
20	150	58.0683	61.29	4.60
30	150	57.8321	61.45	4.61
60	150	57.9375	61.38	4.60
90	150	57.8218	61.45	4.61
120	150	57.6382	61.57	4.62

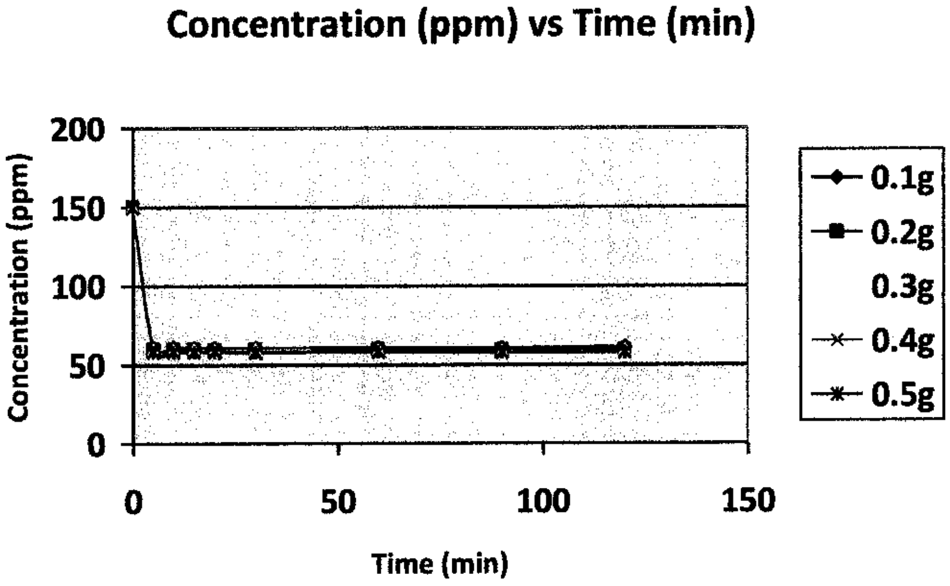

Figure 21: Effect of contact time on concentration of Ni(II) ion solution

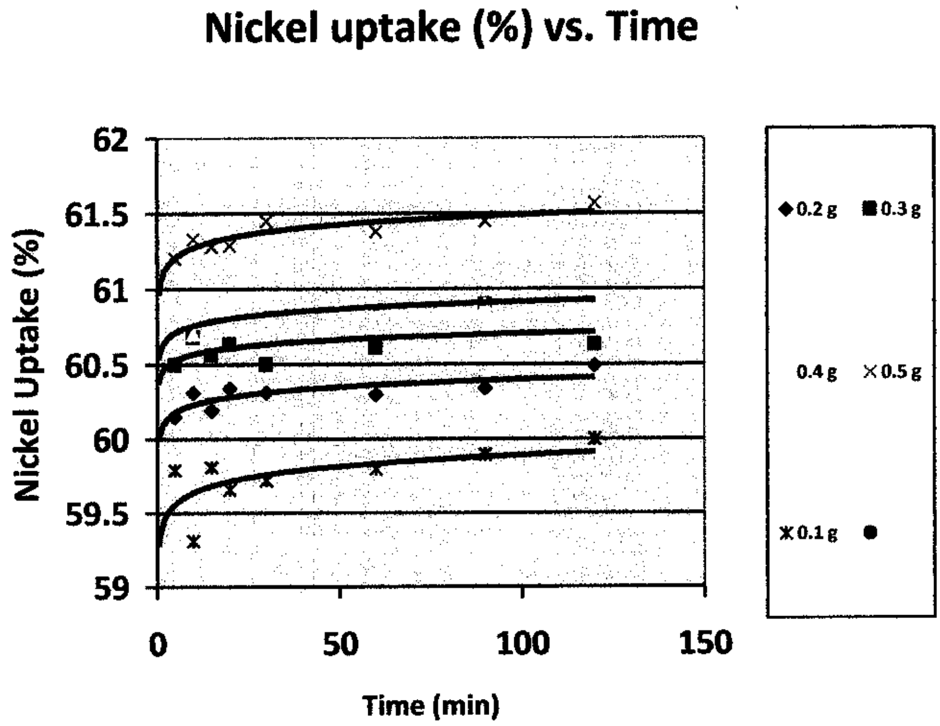

Figure 22: Effect of contact time on percent Nickel uptake

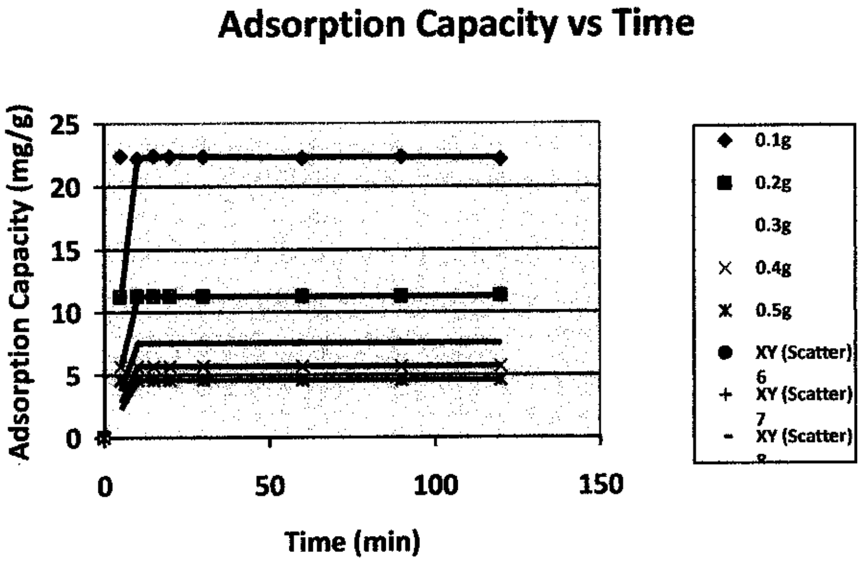

Figure 23: Effect of contact time on adsorption capacity

Based on the results obtained, it is known that the concentration decreased as the loading is increased. It is also known that adsorption capacity increased as the contact time increased. As shown from the figure, rate of adsorption was fast in the initial stage and only change slightly as the adsorption extent to the respective period.

4.6 ISOTHERM STUDIES

Experimental isotherm is useful for describing adsorption capacity. The relationship between the amount of a substance adsorbed at constant temperature and its concentration in the equilibrium solution is called the adsorption isotherm

4.6.1 Langmuir isotherm

Langmuir equation:

$$1/q = 1/Q_{max} + 1/(Q_{max}bC_e)$$

Ce is the concentration of the metal solution at equilibrium (mg/l)
Q is the amount of metal adsorbed at equilibrium (mg/g)
Q_{max} is the maximum sorption capacity of the metal-rice husks system.
b is constant related to the binding energy of the sorption system.

For this isotherm model, graph of 1/q versus Ce was plotted to observe the trending for adsorbent prepared at different loading of carbonized rice husk that carbonized at 500°C

Table 15 : Experimental data for Langmuir Isotherm

Loading (g)	$1/q = 1/Q_{max} + 1/(Q_{max}bC_e)$		
	Q_e	$1/Q_e$	C_e
0.1	22.24	0.04496	61.0317
0.2	11.31	0.0884	59.5281
0.3	7.59	0.13175	58.976
0.4	5.69	0.1757	58.9501
0.5	4.6	0.21739	57.9974

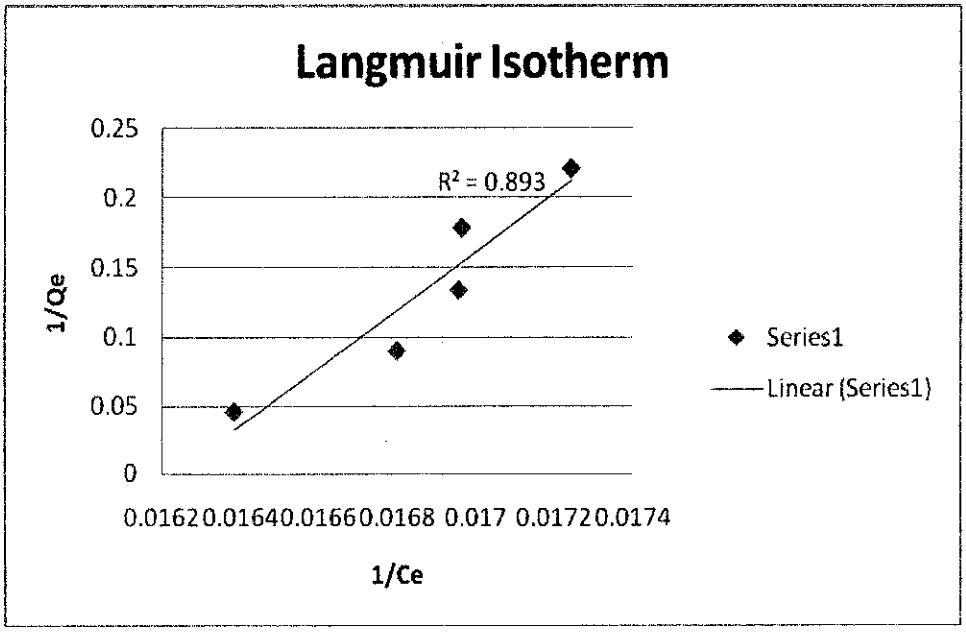

Figure 24: Langmuir Isotherm Plot for Rice Husks Carbonized at 500°C

4.6.2 Freundlich isotherm

Freundlich equation:

$$\text{Log } q = 1/n \log C_e + \log K$$

Ce is the concentration of the metal solution at equilibrium (mg/l)

Q_e is the maximum sorption capacity of the metal-rice husks system.

K and 1/n are constant ,which are considered to be the relative indicators of adsorption capacity and adsorption intensity.

From Freundlich isotherm model, graph log q versus log C_e was plotted to observe the trending for adsorbent prepared at different loading of activated carbon carbonized at 500°C.

Table 16 : Experimental data for Freundlich Isotherm

Loading (g)	$\text{Log } q = 1/n\log C_e + \log K$	
	Log Q_e	Log C_e
0.1	1.347135	1.785555
0.2	1.053463	1.774722
0.3	0.880242	1.770675
0.4	0.755112	1.770485
0.5	0.662758	1.763409

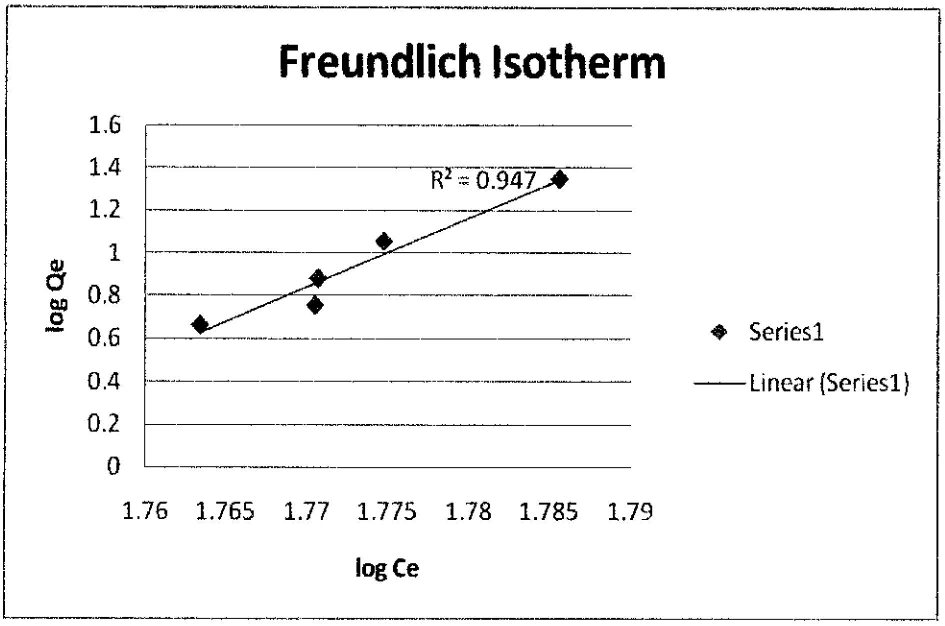

Figure 25: Freundlich Isotherm Plot for Rice Husks Carbonized at 500°C

Based on the R^2 obtained, it is known that this adsorption of rice husk is suits for Freundlich isotherm as the amount is 0.947 which is higher compared to Langmuir isotherm which is 0.893.

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSIONS

Based on the results obtained throughout the study,several conclusions can be made:

1. The pores of rice husk are developed through chemical treatment with sodium hydroxide and carbonization process; and highly porous rice husk based activated carbon with high adsorption capacity was produced. Thus, we can conclude that adsorption also increased as the loading is increased,until it reached equilibrium.
2. Abundantly available rice husk give the potential as an alternative to current industry activated carbon for removal of Ni (II) ion from aqueous solution and can be utilized for waste water treatment.

5.2 RECOMMENDATIONS

Several recommendations have been identified to further improve the current research for future use.

1. The siever need to have some shaking resistance as it tends to move out from the position if the magnitude of shaking is high.
2. The grinder should be replaced as it is not optimally functioning as it is before. This increase the time of the preparation process.
3. To study the effect of solution pH on adsorption activities.
4. To study the effect of initial concentration of aqueous solution on adsorption activities by varying initial concentration of Ni(II) ions solution.
5. Adsorption from rice husk can be as an alternative to current industrial activated carbon. Hence, it will be great if UTP ventured with the company for them to practice using rice husk in their wastewater treatment.

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APPENDICES

APPENDIX I

Calculation of 150 ppm Ni(II) ions solution

In order to minimize error, the solution prepared is started from 500 ppm $NiCl_2$

500 ppm by mass = 500 mg Ni(II) / 1 kg of water

Given molar mass: Ni - 58.6934 g/mol $NiCl_2$ -129.5994 g/mol

Assuming 1.0 mol of $NiCl_2$, thus having 1.0 mol of Ni(II)

Mass of Ni(II) ions = (58.6934 g Ni(II) X 1 mol of Ni(II)) / 1 mol of Ni(II)

= 58.6934 g Ni(II)

$$\text{Mass of } NiCl_2 = \frac{129.5994 \text{ g } NiCl_2 \text{ X } 1 \text{ mol } NiCl_2}{1 \text{ mol } NiCl_2}$$

$$= 129.5994 \text{ g } NiCl_2$$

$$\text{Percent mass of Ni(II)} = \frac{58.6934 \text{ g Ni(II)}}{129.5994 \text{ g } NiCl_2} = 0.45288 \text{ g Ni(II)/g } NiCl_2$$

Then, 500mg of Ni(II) ions in $NiCl_2$;

$$500 \text{ x } 10^{-3} \text{ g of Ni (II) x } 0.45288 \text{ g Ni(II)/g } NiCl_2 = 0.22644 \text{ g of } NiCl_2$$

By using dilution formula, $\mathbf{M_1V_1=M_2V_2}$

$500V_1=150(1000ml)$ → $V_1 = 300$ ml

Therefore, **300 ml of 500ppm of Ni (II)** ions solution is diluted to prepare 150 ppm of Zn(II) ions

APPENDIX II

Calculation of percent metal uptake

The calculation for metal uptake is such as below:

$$\frac{\text{Initial concentration} - \text{final concentration}}{\text{Initial concentration}} \times 100\ \%$$

For example the initial concentration is 150 ppm and the final is 60 ppm, thus the percent metal uptake is

$$\frac{150\ \text{ppm} - 60\ \text{ppm}}{150\ \text{ppm}} \times 100\%$$

$$= 60\%\ \text{metal ion uptake}$$

Calculation for adsorption capacity

The calculation for adsorption capacity is such as below:

$$\frac{\text{Initial concentration} - \text{final concentration}}{\text{Weight of rice husk (g)}} \text{ X Volume of Solution (L)}$$

For example, the initial concentration is 150 ppm and the final concentration is 60 ppm,volume of solution is 0.015 L and weight of the rice husk activated carbon used is 0.1g, therefore the adsorption capacity is:

$$\frac{150\text{ppm} - 60\text{ ppm}}{0.1\text{g Rice Husk}} \text{ X } 0.015\text{L}$$

$$= 13.5 \text{ mg Ni(II)/g rice husk}$$

APPENDIX III

Figure 26: Raw Rice Husk

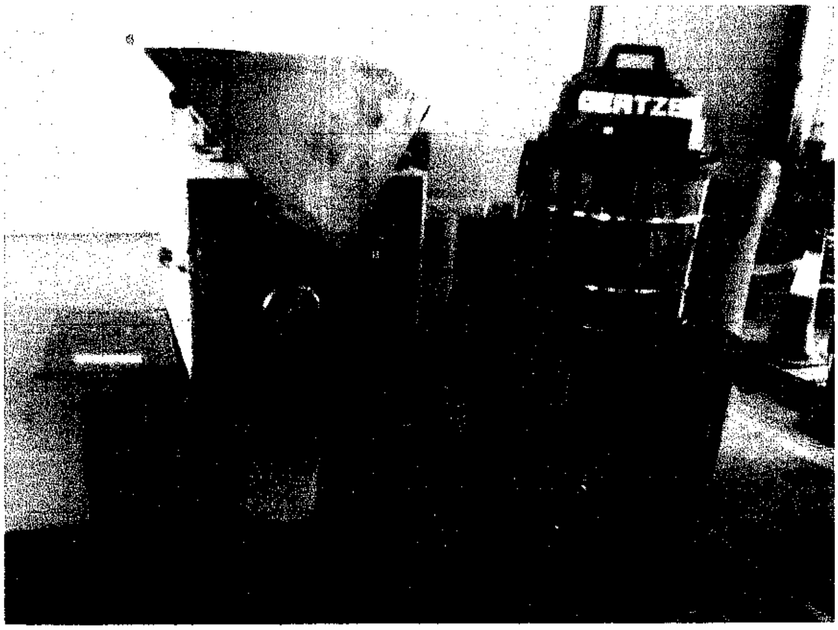

Figure 27: Grinder

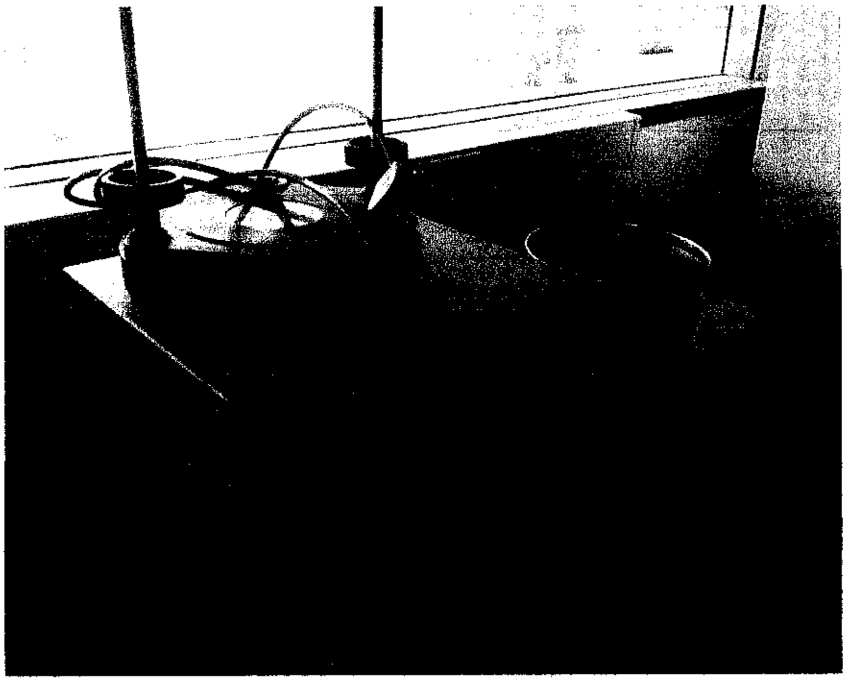

Figure 28: Siever

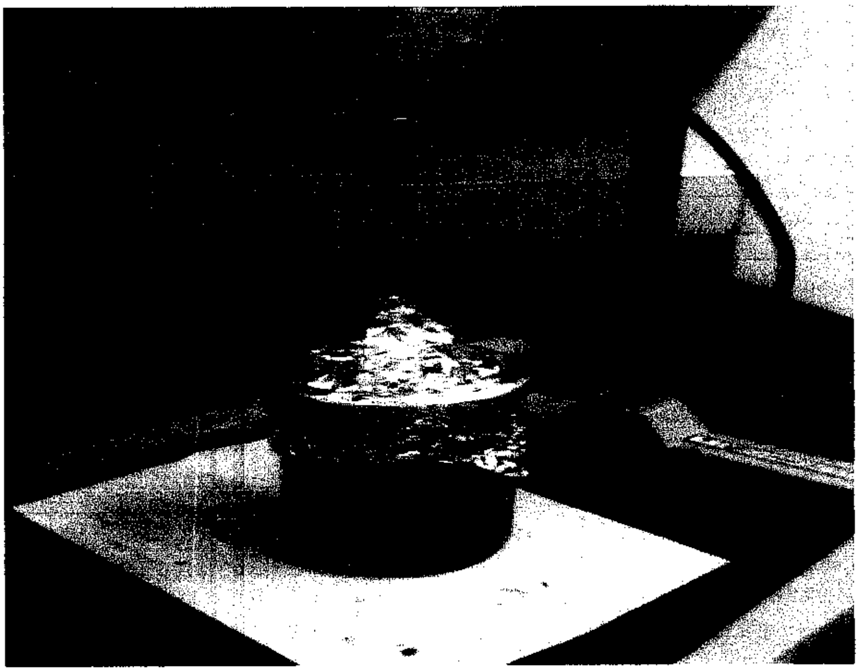

Figure 29 : Treatment Process

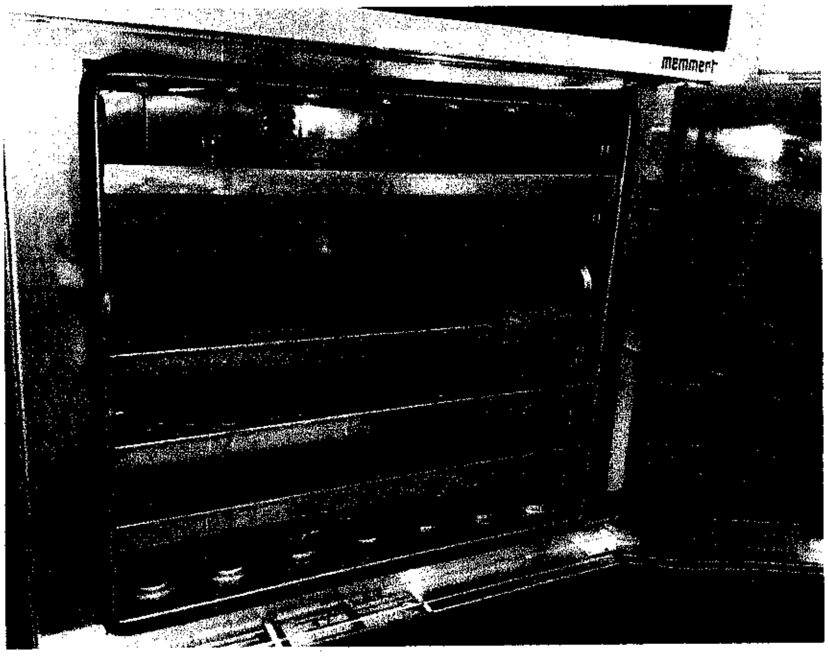

Figure 30: Oven for Drying Process

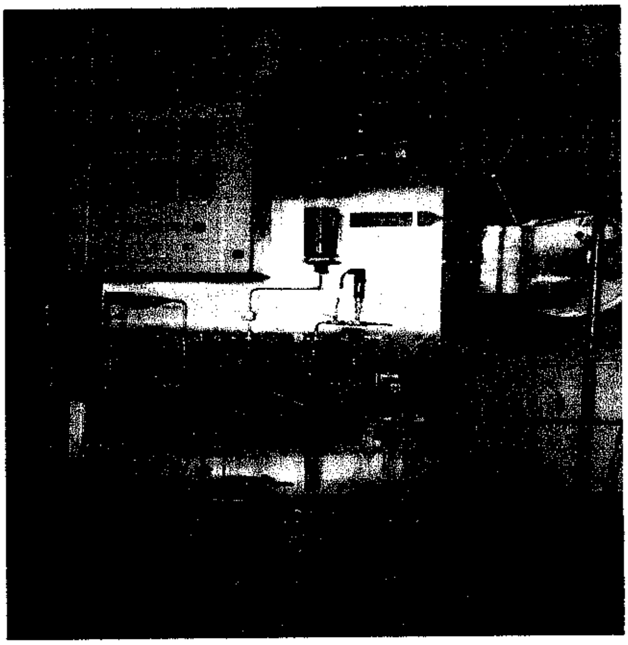

Figure 31 : Furnace with Nitrogen Flow

Figure 32: Carbonized Rice Husk

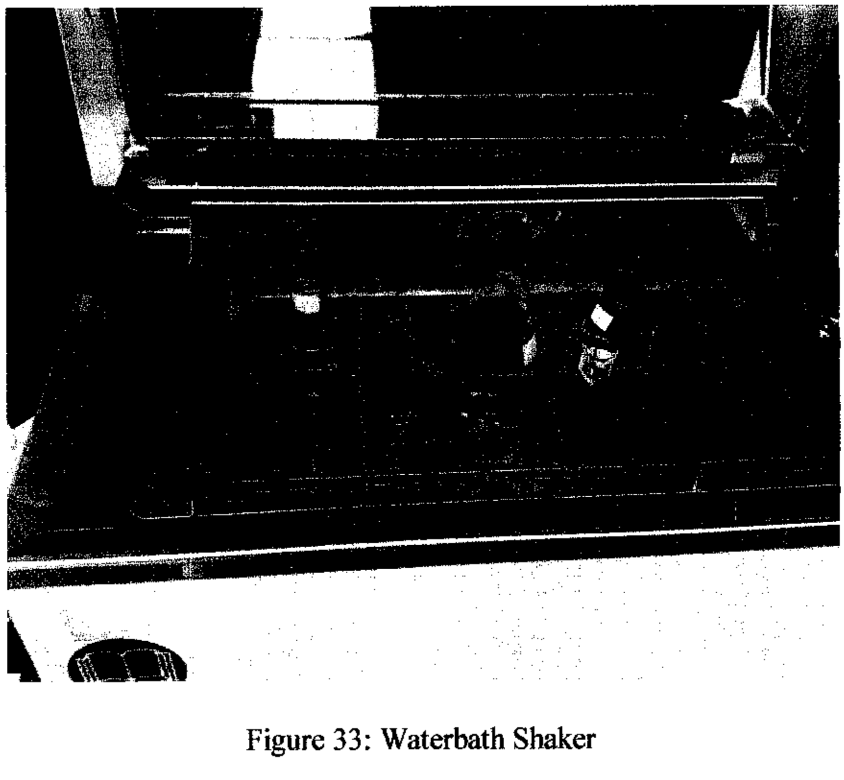

Figure 33: Waterbath Shaker