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TEKNOLOGI
PETRONAS

**Adsorption of Heavy Metal (Nickel) In Waste Water Treatment
Using Banana Peels**

by

Nurul Hidayah binti Mohamed Ghozali

13985

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

MAY 2012

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,



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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.



(NURUL HIDAYAH BINTI MOHAMED GHOZALI)

ABSTRACT

Heavy metal contamination exists in aqueous wastes of many industries in Malaysia and nickel is of the many type of heavy metal ions that are available in the Malaysian waste water streams. There are various ways to remove heavy metal ions from waste water; however the main disadvantages such as economically unattractive and ineffectiveness of the methods has led to the development of alternative technologies. Adsorption is being widely used for removal of heavy metals from waste streams and activated carbon has been frequently used as an adsorbent. However, agricultural wastes are currently being experimented as adsorbents which here in Malaysia, are in abundant and cost effective. Banana peels have been more popularly used in many studies as being one of the more effective and inexpensive type of agricultural waste. This project aims to study the adsorption capacities of three different types of banana peels as an adsorbent to adsorb nickel by manipulating variables such as the contact time, dosage of banana peels and nickel concentrations at constant room temperature. The banana peels' surface characteristics were studied using the CHN Analyzer and the FTIR (Fourier Transform Infrared) spectrometer. Furthermore, the batch adsorption tests were analyzed using the AAS Analysis.

ACKNOWLEDGEMENT

First of all, praise to the Almighty for His blessing on me to carry out and complete Final Year Project (FYP II) project for May 2012 Semester. I am very grateful to finish the project within the time given and complete FYP course for this semester. A tremendous amount of appreciation and gratitude I express towards my beloved and dedicated supervisor Prof Ir. Abdul Aziz Omar for his guidance and advice, lessons and experiences that he shared and taught me throughout this semester throughout the project completion period. Without any doubt he really helped me during the completion of the project. Of all, I would like to thank the Chemical Engineering Department generally for the opportunities to perform the project successfully.

Apart from that, I am very thankful to my family and fellow friends who gave moral support to motivate and allows me to pursue to greater heights in our project. Last but not least, I would also like to thank again those who have directly or indirectly involved in this project as I could not do the project without those assistance and support.

Thank you.

Regards,

Nurul Hidayah binti Mohamed Ghozali

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

As cited from Khan et al.,(2004), he mentions that on a global scale, excessive release of heavy metals into the environment due to the industrialization and urbanization has posed a problem. Unlike organic pollutants that are mostly susceptible to biological degradation, heavy metal ions do not degrade into environmentally friendly end products. There are now at least 20 metals which are classified as toxic they are being emitted into the environment in quantities that pose serious risks to human health (Nomanbhay and Palanisamy, 2005). Basically, heavy metal contamination exists in aqueous wastes of many industries, such as metal plating, mining operations, tanneries, chloroalkali, radiator manufacturing, smelting, alloy industries and other related industries (Kadirvelu et al., 2001). He also stated that some of the most dangerous metals are nickel, zinc, chromium, iron, manganese, mercury, cadmium and lead. Moreover, heavy metal ions such as nickel specifically enters the environment as a result of human activities such as mining purifying, lead and cadmium ores, steel production, coal burning, galvanizing from waste water of pharmaceuticals, paints, pigments, insecticides, cosmetics and from other related industries (Bhattacharya et al., 2006).

Kaewsarn et al.,(2008) discusses on the treatment processes for heavy metal removal from waste water which include adsorption, chemical precipitation, membrane filtration, ultra-filtration, reverse osmosis, electrochemical dispersion and ion exchange. According to Kurniawan et al., (2005), adsorption is a mass transfer process by which a substance is transferred from a liquid phase to the surface of a solid, and becomes bounded by physical and/or chemical interactions. On a large scale, adsorption of heavy metal ion is extensively used as a major unit operation in industrial waste water treatments. Furthermore, numerous studies have shown that activated carbon has been widely and efficiently used as an adsorbent. However, despite its extensive use in waste water treatment industries, they remain to be an expensive material. Therefore in recent years, the need for alternative methods that are safe and economical for elimination of heavy metals from contaminated waters

has necessitated research interests towards the production of low cost alternatives to activated carbon (Nhapi and Banadda, 2011).

According to Memon et al.,(2008), many efforts have been carried out to identify new sorbents for the removal of heavy metals which are both effective and economical. Adsorption via wastes such as the banana peels is currently widely being used as one of the best adsorbent based on their characteristics that we will see in this research later.

1.2 Problem Statements

Main source of drinking water comes from groundwater and unfortunately most of the water sources even here in Malaysia are highly contaminated. Nickel is only one of the many serious heavy metal contaminants that are classified as toxic. Technically, heavy metal ions can easily penetrate and seep into groundwater sources, contaminating the stream flow (Memon et al., 2008). Hence, the challenge is to maintain safe drinking water which is free from heavy metals as they can affect human's health. Moreover, Memon et al.,(2008) also adds that long term effects of drinking water with high levels of contaminants could lead to skin, kidney and lung cancer, neurological disorders, muscular weakness, loss of appetite and nausea. On the other hand, the abundance of agricultural waste also creates a problem as they are classified as waste and disposing them off requires capital as well. Thus, if such wastes can be used as means to treat waste water it will help to lessen the burden of agricultural waste disposal. Due to the ineffective and high cost of current waste water treatments, researchers are finding alternative methods that can remove heavy metals from waste water streams. Currently, the new method focuses on using agricultural by-products that are cheap and in abundance here in Malaysia. For this research, the author has decided to apply the adsorption technique to remove heavy metals using banana peels as the adsorbent to adsorb the nickel ions.

1.3 Objectives and Scope of Study

The main objective of this research is to study the adsorbing capacities of three different types of banana peels as an alternative adsorbent by adsorbing nickel ions as the heavy metal in order to determine the best species of banana for the adsorbent.

Moreover, this research seeks to study the effect of the time variable such as the contact time between the banana peels and a manganese solution with specific concentration by finding out the time required to reach equilibrium. The type of banana peels that has the highest percentage of nickel removal efficiency within equilibrium time will be the best type of adsorbent. Also, the author also aims to study the effect of adsorbent (banana peels) dosage on adsorption activities. The type of banana peels that is able to adsorb the highest amount of nickel with the smallest amount of adsorbent will be deemed as the best choice of adsorbent. The final variable that would be experimented on is the concentration of nickel solution. The author will be able to determine the best type of banana peels because the type that is able to remove nickel ions at high concentrations will be the best type of adsorbent. Also, the adsorption isotherm is important in this research and therefore the author will analyze the adsorption isotherms for both Langmuir and Freundlich model. The objectives of this research are summarized as below:

- To study the capabilities of different type/species of banana peels as an adsorbent to adsorb nickel (heavy metal).
- To study the effect of contact time on adsorption activities.
- To study the effect of adsorbent dosage (banana peels) on adsorption activities.
- To study the effect of heavy metal (nickel) concentration on adsorption activities.
- To study the isotherm model for the adsorption activities.

CHAPTER 2

LITERATURE REVIEW

2.1 Heavy Metals Contamination in Malaysia

Onundi et al., (2010) highlighted that high concentration of heavy metals in the environment can be seriously dangerous to a variety of living species, especially human beings. This is because excessive ingestion of these metals by humans can cause accumulative poisoning, cancer, nervous system damage and ultimately death. Major sources of heavy metals contamination comes from industries dealing in electroplating, electronics, batteries and metal treatment/fabrication. Many of these industries are located in the western coast of the peninsular Malaysia, which includes Klang Valley, Malacca, Johor Bahru and Penang areas. Today, there are many increased concerns by environmentalists and governments on the effects of heavy metals and therefore, efforts in attempting to protect public health has resulted in increased research in the development of advance technologies to remove heavy metals from waters and wastewaters (Onundi et al.,2010).

Water pollution is a serious problem in Malaysia and impacts negatively on the sustainability of water resources. For instance, in gas and petrochemical sectors alone, the wastewater from the area primarily contains heavy metal ions such as Nickel (Ni), Mercury (Hg), Cadmium (Cd), Zinc (Zn), Chromium (Cr), Iron (Fe), Lead (Pb) and Copper (Cu) (Kodori et al., 2010). She also adds that pollution reduces total water availability in some cases; due to high costs, polluted waters could become non treatable for consumption. In addition, water pollution is caused by both point-sources and non-point sources. Point sources include sewage treatment plants, manufacturing and agro-based industries and animal farms. On the other hand, nonpoint sources are mainly diffused sources such as agricultural activities and surface runoffs (Kodori et al., 2010). Figure 1 shows the composition of water pollution sources by sector in Malaysia and Figure 2 demonstrated the distribution of water pollution sources by states. This was recorded by the Department of Environment (DOE) in 2008.

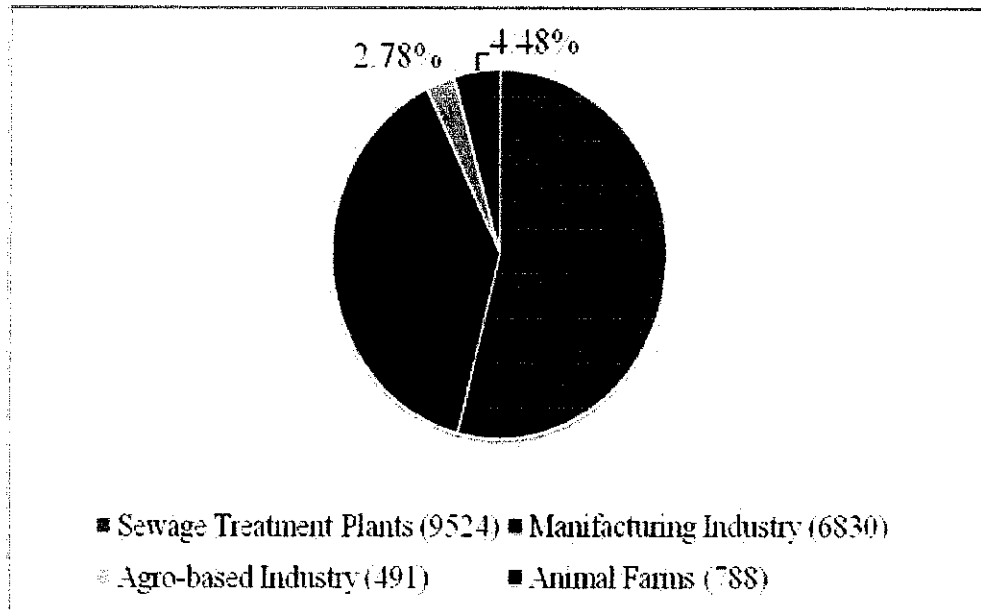


Figure 1: Malaysia Composition of water pollution sources by sectors (DOE 2008)

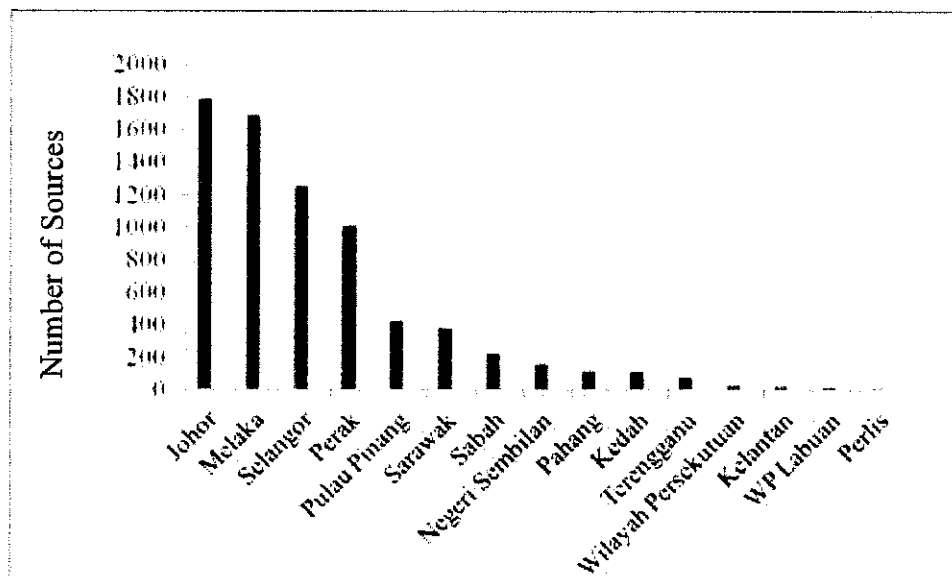


Figure 2: Distribution of Industrial Water Pollution Sources by States (DOE 2008)

2.1.1 Impact of Heavy Metals

Generally, heavy metals can cause various diseases and disorders as they are not biodegradable and tend to accumulate in living organisms (Wang et al., 2003). Due to their level of toxicity, bio-accumulating tendency and threat to human life and the environment, heavy metals is no doubt a major concern. Furthermore, heavy metals are not subjected to bacterial attack or other break down or degradation process and therefore become a permanent addition to the environment. Hence in time, they will find their way up the food pyramid. As this occurs and they accumulate in the

environment and in food chains, they can profoundly disrupt biological processes (Igwe and Abia, 2006). Moreover, Volesky (1990) stresses that excessive amounts of a metal species can indeed lead to the disruption of cellular functions and even death (Volesky, 1990). Thus, since most of heavy metals are non-degradable and toxic, the realistic option is to reduce the concentration to acceptable levels before discharging them into the environment (Calace et al., 2002).

According to Kurniawan et al., (2005), to achieve an economically effective treatment of metal-contaminated wastewater, various low-cost materials have been investigated worldwide, such as in India, Thailand, Nigeria, Italy and USA. The reported maximum contaminant level (MCL) of heavy metals in surface water and their toxicities are as shown below in Table 1.

Table 1: Maximum contaminant level (MCL) of heavy metals and their toxicities

		USA EPA (2004)	Hong Kong EPD (2004)	Thailand PCD (2004)
Cr (VI)	Headache, nausea, diarrhea,	0.05	0.05-0.10	0.25
Cr (III)	vomiting	0.10	-	0.75
Zn (II)	Depression, lethargy, neurological signs such as seizures and increased thirst	1.00	0.60-1.00	5.00
Cu (II)	Liver damage, insomnia	0.25	0.05-0.10	2.00
Cd (II)	Kidney damage, renal disorder	0.01	0.001-0.05	0.03
Ni (II)	Dermatitis, nausea, chronic asthma, coughing	0.20	0.10-0.20	1.00

Remarks:

*Environmental Protection Agency (EPA), USA

*Environmental Protection Department (EPD), Hong Kong Administrative Region (HKSAR)

*Pollution Control Department (PCD), The Ministry of Natural Resources and Environment, Thailand

2.2 Existing Technologies for Removal Mechanisms

Wang et al., (2003) wrote that the treatment methods for metal-bearing effluents commonly include chemical precipitation (hydroxides, sulfides, etc.), membrane filtration (reverse osmosis, nanofiltration, etc.), electrolytic reduction, solvent extraction, ion exchange, and adsorption. However, like any other technologies, there are of course some advantages and disadvantages. For instance, the removal of heavy metals by reverse osmosis (RO) is possible, but limitations such as the fouling of membranes and the need for pre-treatment should be considered. Also, RO requires a high operating pressure. Moreover, solvent extraction and electrolytic processes are also available but they are considered to be economical only for more concentrated solutions. The cost-effective alternative techniques or materials for removal of heavy metals from diluted solutions are hence needed. Next is the liquid-phase adsorption which is one of the most efficient methods but is only effective for the removal of colors, odors, organic, and inorganic pollutants from industrial effluents.

Finally, granular or powdered activated carbon is the most widely used adsorbent because of its ability to remove relatively low-molar-mass organic compounds. However, activated carbons are only effective when physical and chemical modifications have been made. In this case, their use is usually limited due to their high cost. Therefore, this problem has led to the search for cheaper adsorption substitutes including fly ash, lignin, peat, natural materials (chitosan, alginate, biomass, etc.), and clays (clinoptilolite, montmorillonite, kaolinite, etc.) (Wang et al., 2003). In the last few years many studies have been focused on identifying inexpensive but effective adsorbents (Calace et al., 2002).

2.2.1 Emerging Technologies

Natural materials that are available in large quantities from agricultural operations have great potential to be used as low cost adsorbents, as they represent unused resources, widely available and are environmentally friendly (Deans and Dixon, 1992). For example, in Malaysia, oil palm is the most important commercial crop and it was reported that Malaysia currently produces about 30 million tonnes annually of oil palm biomass, including trunks, fronds, fruit waste and empty fruit branches (Nomanbhay and Palanisamy, 2005). Moreover, peat moss was used for the

removal of mercury, copper, cadmium and nickel; cadmium and zinc were adsorbed on waste tea leaves. In addition, soils and clays have also shown remarkable potential towards the removal of heavy metals ions from water (Awan et al., 2003). Potential agricultural adsorbents include coconut and rice husk, sawdust, sunflower, tea and rubber leaf powder papaya wood, oil palm shell, sugarcane bagasse and other low cost agricultural waste by-products (Wang et al., 2003).

2.2.2 Banana Peel as Adsorbent

Banana peel is an inexpensive and easily available material which can be utilized as an alternative to the more costly adsorbents. In a previous study by Mandi et al. (2010), banana peels was proven to be a promising material for the removal of contaminants (phenol) from olive mill wastewaters. Not only banana peel is an abundant cheap adsorbent, but it is also highly efficient. In the study, the banana peel showed a high adsorption capacity of phenolic compounds (688.9 mg/g), revealing that banana peels could be employed as a promising adsorbent for phenolic compounds adsorption (Mandi et al., 2010). Generally, banana peel is believed to be suitable as biosorbent because of its proven high sorption capacities of heavy metals due to the presence of functional groups like COOH, -OH and amines.

In this research, the author has chosen to utilize three different types of banana namely the 'Pisang Berangan' (*Musa acuminata Colla-AAA Group*), 'Pisang Tanduk' (*M. corniculata Rumphias-AAA Plantain Subgroup*) and 'Pisang Emas' (*M. acuminata* and *M. balbisiana-AA Sucrier Group*) (Ploetz et al., 2007).

All three types of bananas have different physical characteristics as the Pisang Emas is smaller in size compared to the Pisang Berangan and the Pisang Tanduk is the largest one in size and is shaped like a horn. The author will explain further on the selection of bananas based on their banana peels' surface characteristic tests using the CHN Analyzer, BET method and the FT-IR analysis.

2.3 Adsorption

The adsorption process has been found to be a very practical approach to remove heavy metals as proven in various previous studies using activated carbon. Adsorption using commercial activated carbon (CAC) can remove heavy metals from wastewater, such as Cd, Ni, Cr and Cu. However, CAC remains an expensive material for heavy metal removal (Nomanbhay and Palanisamy, 2005). The existence of some specific functional groups might be associated with the capability of carbon to adsorb heavy metals.

The process of adsorption is defined as a process of collecting substances in water onto a surface of a solid. Another extended definition of adsorption is the process of accumulating substances that are contained in a solution onto a suitable interface (Metcalf and Eddy, 2004). When a liquid mixture comes into contact with a microporous solid, adsorption takes place. There are several factors that affect the efficiency of adsorption.

According to Metcalf and Eddy (2004) there are two major processes in adsorption that occur which is the physisorption and chemisorption. The functional group which has high affinity to metals will adsorb the metal during physisorption which will last until the equilibrium state. Time required in achieving the equilibrium time (t_e) and the concentration at t_e is called equilibrium concentration (C_e). The residual metal concentration which is not adsorbed during physisorption will be adsorbed by chemisorption process. The chemisorptions process is described as the intrapore diffusion of heavy metals into carbon molecules.

However, there are some chemical and physical modifications that have to be applied to activate the carbon surface. In physical modification, the carbon would have to be heated up until 750°C to sustain combustion. Then, it will have to be exposed to oxidizing gas such as steam and CO₂ at high temperature in the range of 800°C to 900°C. However, in the chemical modification method, formaldehyde, nitric acid (HNO₃) and pH controls the solution which is a combination of sulphuric acid (H₂SO₄) and sodium hydroxide (NaOH).

2.3.1 Adsorption Isotherm

In developing the adsorption isotherm, the quantity of adsorbate that can be taken up by an adsorbent is a function of both characteristics and concentration of adsorbate and the temperature. Generally, the amount of material adsorbed is determined as a function of the concentration at a constant temperature, and the resulting function is called an adsorption isotherm (Metcalf and Eddy, 2004).

Equations that are often used to describe the experimental isotherm data were developed by Freudlich, Langmuir, and Brunauer, Emmet and Teller (BET) isotherms. One of the three which is the Freudlich isotherm is used most commonly to describe the adsorption characteristics of the activated carbon used in water and wastewater treatment. Derived empirically in 1912, the Freudlich isotherm is defined as below:

$$x/m = K_f C_e^{1/n} \quad \text{Equation (1)}$$

- x/m : Mass of adsorbate adsorbed per unit mass of adsorbent, mg adsorbate/g activated carbon
- K_f : Freudlich capacity factor, (mg adsorbate/g activated carbon) (L water/mg adsorbate)^{1/n}
- C_e : Equilibrium concentration of adsorbate in solution after adsorption, mg/L
- $1/n$: Freudlich intensity parameter

The following is the Langmuir isotherm published by Irving Langmuir in 1916 for gases adsorbed to solids. It is a semi-empirical isotherm derived from a proposed kinetic mechanism. The constant can be written as:

$$\text{Log } (x/m) = \text{log } K_f + 1/n \text{ log } C_e \quad \text{Equation (2)}$$

Derived from rational consideration, the Langmuir adsorption isotherm is defined as:

$$x/m = abC_e / (1 + bC_e)$$

- x/m : Mass of adsorbate adsorbed per unit mass of adsorbent, mg
adsorbate/g banana peels
- a,b : Empirical constant
- C_e : Equilibrium concentration of adsorbate in solution after
adsorption,mg/L

Equation (2) above is based on four assumptions:

- The surface of the adsorbent is uniform, that is, all the adsorption sites are equivalent.
- Adsorbed molecules do not interact.
- All adsorption occurs through the same mechanism.
- At the maximum adsorption, only a monolayer is formed; molecules of adsorbate do not deposit on other, already adsorbed molecules of adsorbate, only on the free surface of the adsorbent

(Metcalf and Eddy, 2004).

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Preparation Procedures

3.1.1 Preparation of adsorbent (banana peels)

The three types of banana peels were obtained from a local market in Ipoh, Perak. Around 1 to 2 bunches of banana (Pisang Berangan, Pisang Emas and Pisang Tanduk) were required to obtain enough samples for the batch experiments. Next, the banana peels were finely sliced, washed thoroughly and dried in an oven at a temperature of 100°C for 48 hours as it is essential to ensure that no moisture remains in the samples. Then, the dried banana peels was grinded using a 'Ball Pulverisette Grinder' to form particles of approximately 0.8 to 2.0 mm in size. The grinded banana peels were stored in individual airtight containers and were carefully labeled.

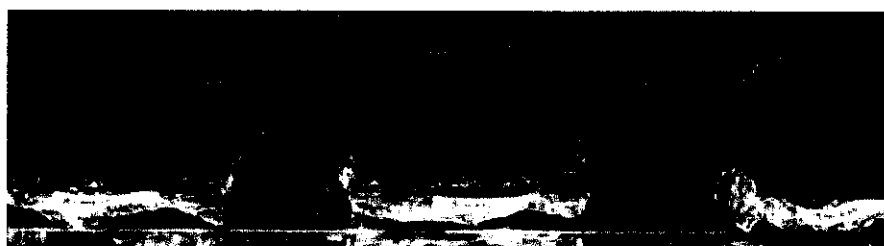


Figure 3: Dried Banana Peels

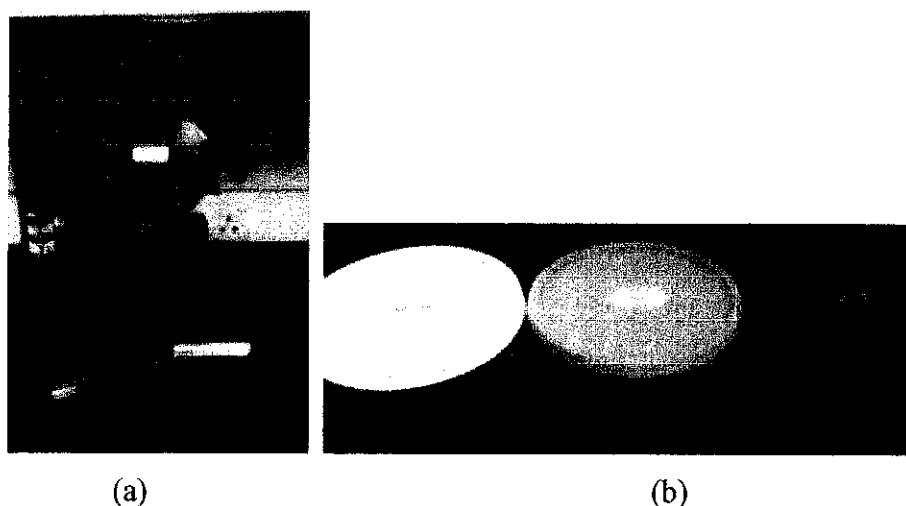


Figure 4: (a) Grinder (b) Air-tight containers to store banana peels

3.1.2 Preparation of nickel solution

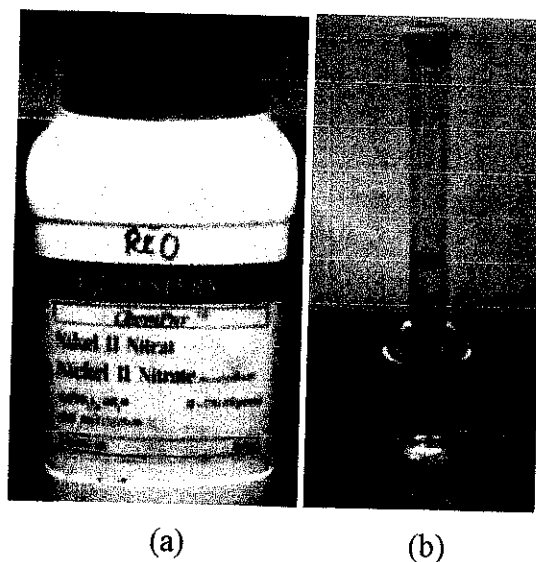


Figure 5: (a) Nickel (II) Nitrate (b) Preparation of solution

Given : MW of $\text{Ni}(\text{NO}_3)_2 = 290.81 \text{ g/mol}$

: MW of $\text{Ni} = 58.6394 \text{ g/mol}$

Thus, dividing the molecular weight with the molecular weight of Ni gives 4.959g of $\text{Ni}(\text{NO}_3)_2$. Hence, using this weight of $\text{Ni}(\text{NO}_3)_2$ and dissolving it in 1 L volume of distilled water will give a 1000 ppm concentration of nickel solution. However, a 2 L volumetric flask was more appropriate because the weight of $\text{Ni}(\text{NO}_3)_2$ required was too small for a 1 L volumetric flask. Using the $M_1 V_1 = M_2 V_2$ formula, a set of five different concentration of $\text{Ni}(\text{NO}_3)_2$ (from the 1000ppm) will be prepared which are 10, 20, 30, 40 and 50 ppm as shown in the table below:

Table 2: Mass of $\text{Ni}(\text{NO}_3)_2$ required

	10ppm	20ppm	30ppm	40ppm	50ppm
	0.1 g	0.198 g	0.298 g	0.396 g	0.496 g

3.2 Surface Characteristic Tests for Banana Peels

The banana peels' surface will be characterized by diffusing reflectance infrared Fourier Transform Infra-Red (DRIFT) using a spectrometer using 200 scans, resolution of 4cm^{-1} and ambient atmosphere. In this procedure, a crucible, used in a reflectance apparatus, was filled with the banana peels particles without dilution. Specific surface area and pore size distribution will be determined with 2.0g of minced banana peel. Besides FT-IR, the sample was also analyzed using a CHN analyzer to determine the composition of carbon, nitrogen and hydrogen of the banana peels. All three types of banana peels' surface characteristic tests was studied. Moreover, the Brunauer Teller Method (BET) will also be conducted to study the individual pore characteristics of all the banana peels.

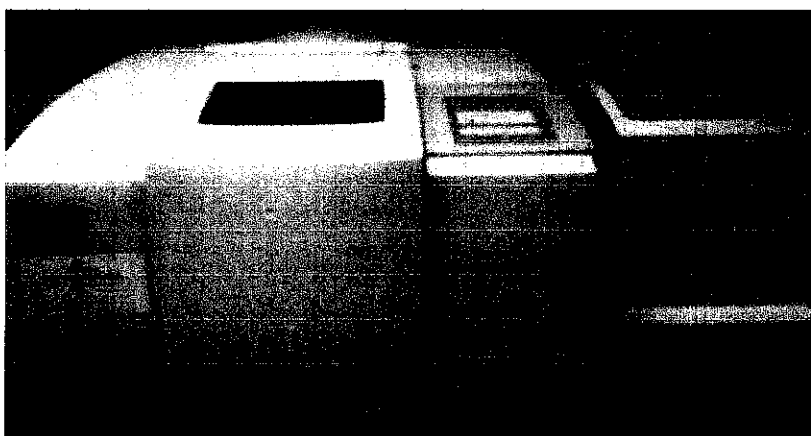


Figure 6: FT-IR Analyzer

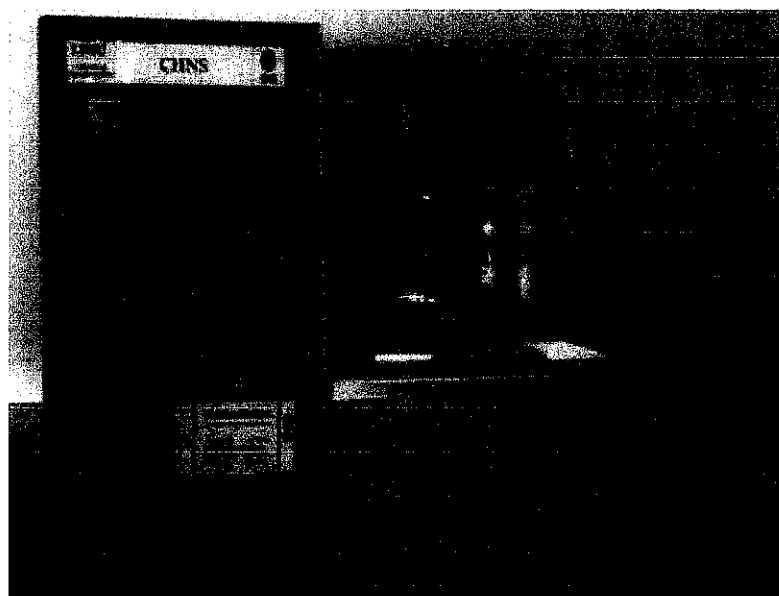


Figure 7: CHN Equipment

3.3 Batch Adsorption Studies

The adsorption of Ni^{2+} was investigated in batch equilibrium experiments. Stock solutions of nickel ions were prepared using the obtained standard solutions, in distilled water. There were three experiments with three different variables that were conducted as explained below.

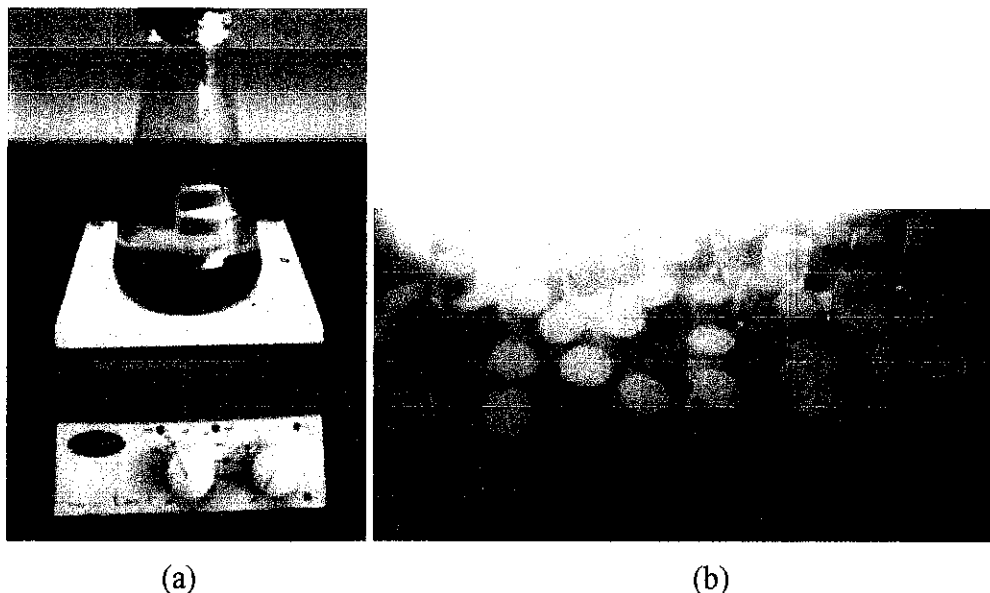


Figure 8: (a) Batch experiment using the magnetic stirrer (b) Test tubes for AAS

3.3.1 Test for contact time

250 mL of Erlenmeyer flasks with 200 ml of adsorbate (Ni) solution was prepared with a concentration of 20 ppm. A mass of 3.0g adsorbent (grinded banana peels) was measured and added to the Erlenmeyer flask. The flasks were then agitated at using a magnetic stirrer using a constant speed of between 150 rpm to 180 rpm since the speed had to be regulated to ensure that there were no spillages. Also, the flasks were covered with aluminium foil to seal the flasks. Then, the adsorbent (banana peels) was removed from the shaker for residual adsorbate analysis at 15, 30, 45, 60, 75, 90, 105 and 120 minutes respectively. The samples were then filtered and the final adsorbate concentrations were measured using the AAS. This experiment was repeated twice for the two other types of banana peels. Using three magnetic stirrers, the experiments were conducted simultaneously.

3.3.2 Test for adsorbent dosage

250 mL of Erlenmeyer flasks with 200 mL of adsorbate (Ni) solution was prepared with a concentration of 20 ppm. The mass of adsorbent (grinded banana peels) of 1.0g, 2.0g 3.0g, 4.0g and 5.0g was measured and added into each of the Erlenmeyer flasks. The flasks were then agitated at around 150 rpm to 180 rpm using a magnetic stirrer. Then they were removed from the shaker for residual adsorbate analysis after 120 minutes. The samples were then filtered and the final adsorbate concentrations were measured using the AAS. This experiment was repeated twice for the two other types of banana peels. Using three magnetic stirrers, the experiments were also conducted simultaneously.

3.3.3 Test for different nickel concentrations

250 mL of Erlenmeyer flasks with 200 mL of adsorbate (Ni) solution was prepared with concentrations of 10ppm, 20ppm, 30ppm, 40ppm and 50ppm. A mass of 3.0g of adsorbent (grinded banana peels) was measured and added into each of the conical flasks. The flasks were then agitated using a magnetic stirrer. Finally, the samples were filtered and the final adsorbate concentrations will be measured using the AAS after 120 minutes. This experiment was repeated twice for the two other types of banana peels and was conducted simultaneously as well.

3.3.4 AAS Analysis

All of the filtered test tubes were analyzed using the AAS (Atomic Absorption Spectroscopy) to check for Ni^{2+} concentration. Graphs were plotted to observe the effects of the variables varied in the batch adsorption experiments.



Figure 9: Atomic Absorption Spectroscopy (AAS)

3.4 List of Equipments and Chemicals

The following is the summarized list of all the equipments and chemicals required for this research.

- Atomic Adsorption Spectrophotometer (AAS)
- Ball Pulverisette Grinder
- Oven
- CHN Analyzer
- Brunauer Teller Method (BET)
- Fourier Transform Infrared Spectroscopy (FT-IR)
- Nickel Nitrate $\text{Ni}(\text{NO}_3)_2$
- Test tubes and rack
- Distilled water
- Erlenmeyer flasks
- 2-L Volumetric flasks

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Banana Peels Analysis

4.1.1 FT-IR Analysis

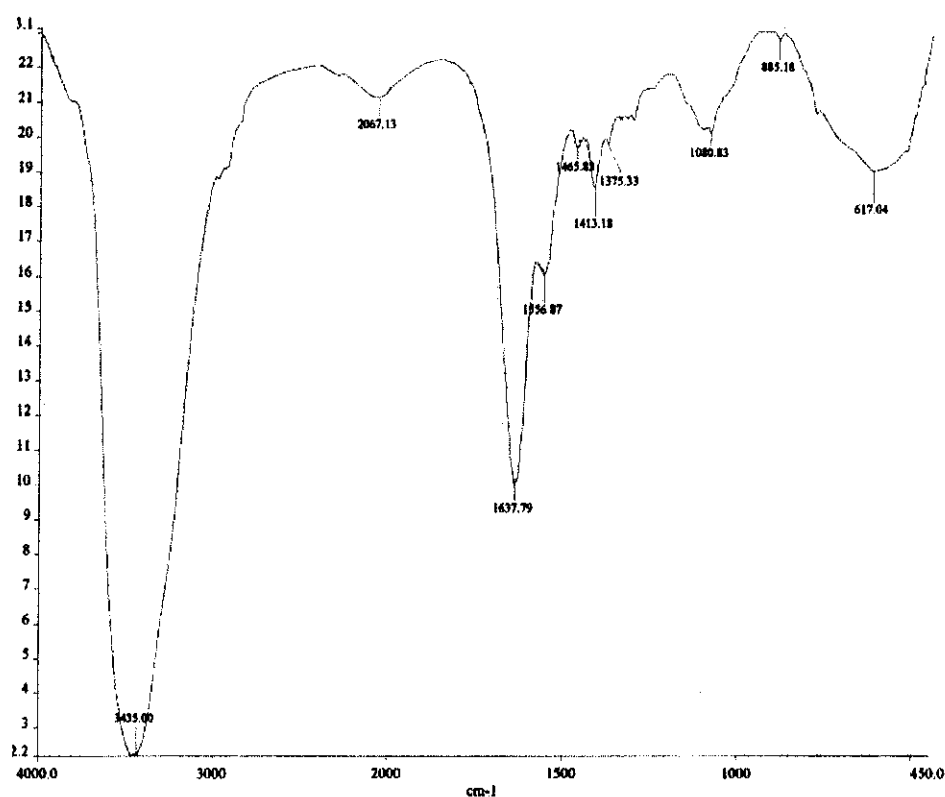


Figure 10: FT-IR of Banana Peels (Pisang Berangan)

FT-IR spectra of banana peel was obtained to identify the functional groups present in the banana peels. FT-IR figure above displays a number of more significant peaks, indicating the nature of the adsorbent. Bands that appear are 3435, 2067.13, 1637.79, 1556.87, 1413.18, 1080.83 and 617.04 cm^{-1} which are carboxylic acids O-H, C-H stretching of alkane, primary amines N-H, alkynes, OH stretching, amide stretch C=O, alkenyl C=C stretch and aromatic C=C bending. Out of these, carboxylic and hydroxyl groups played a major role in the adsorption of Ni^{2+} ions.

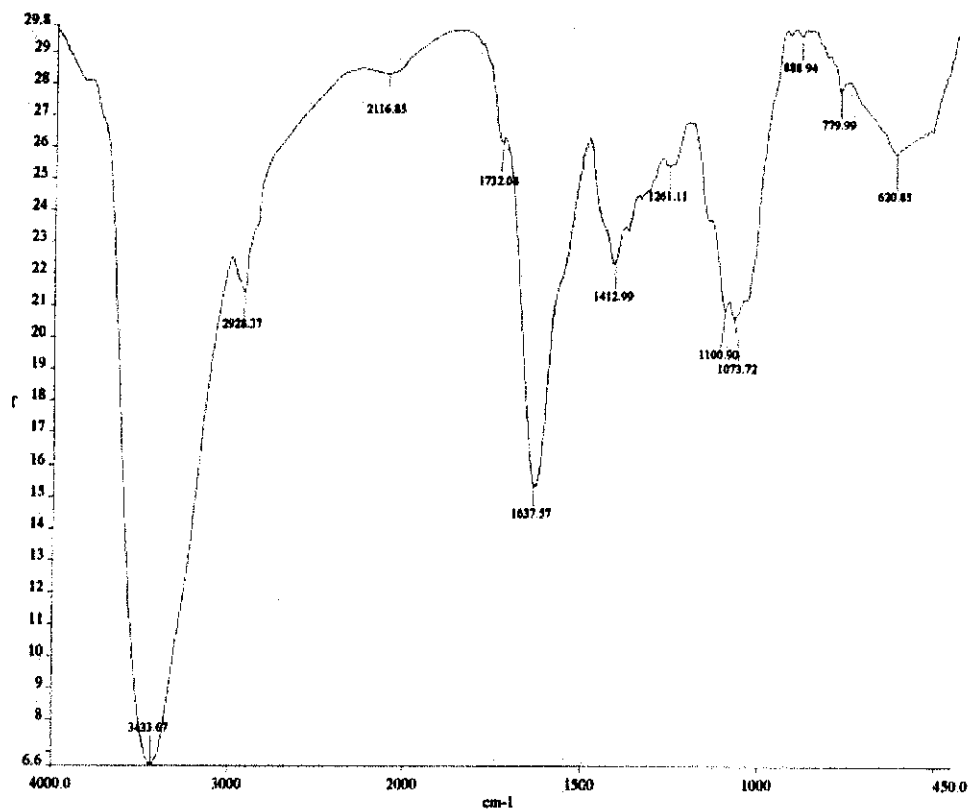


Figure 11: FT-IR of Banana Peels (Pisang Emas)

In Figure 11 above, the more obvious peaks are almost similar with the peaks in Figure 10 except that some of the values slightly vary. The peaks are 3433.67, 2928.37, 1637.57, 1412.99, 1100.90, 1073.72, 779.99 and 620.85 cm^{-1} . These represent alcohol/phenol O-H stretch, amine N-H stretch, aromatic C=C bending, OH stretching, carboxylic acids O-H and C-H stretching of alkane. Similar to Pisang Berangan, carboxylic and hydroxyl groups seems to also play a major role in the adsorption of Ni^{2+} ions.

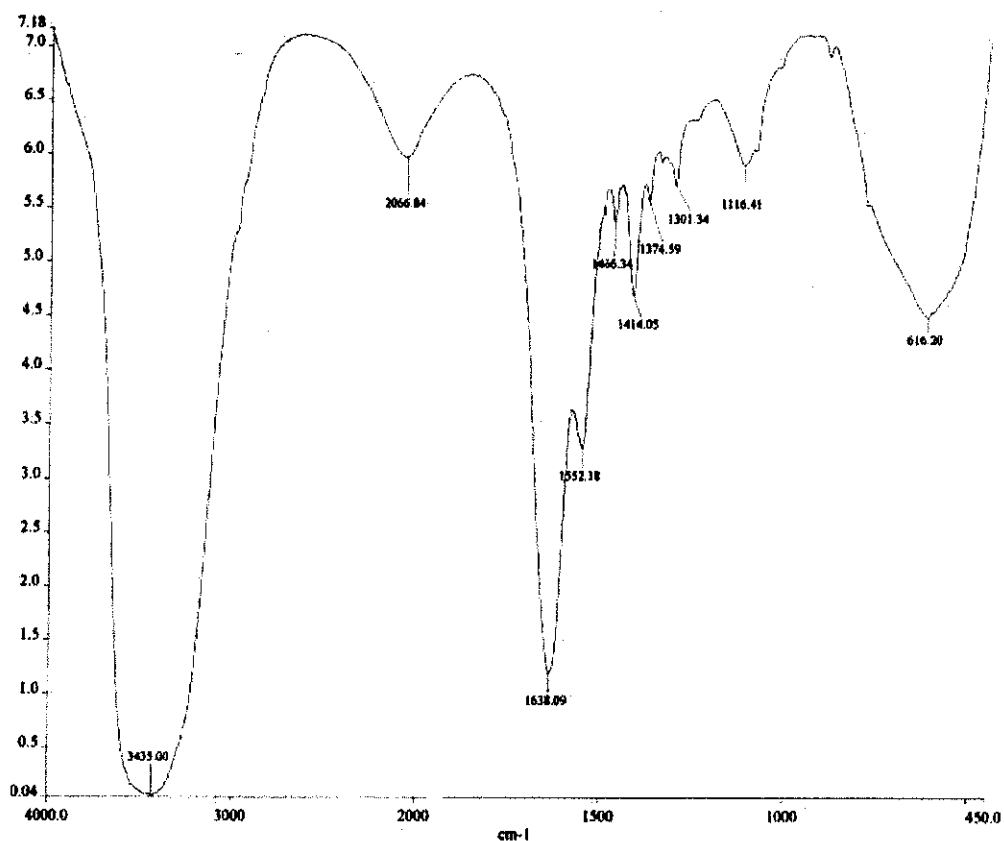


Figure 12: FT-IR of Banana Peels (Pisang Tanduk)

Figure 12 above shows the band peaks in the Pisang Tanduk and the important peaks are 3435, 2066.84, 1638.09, 1552.18, 1414.05, 116.41 and 616.20 cm^{-1} . They are carboxylic acids O-H, C-H stretching of alkane, primary amines N-H, alkynes, OH stretching, amide stretch C=O, alkenyl C=C stretch and aromatic C=C bending which are similar to two of the other types of banana peels. Out of these, carboxylic and hydroxyl groups played a major role in the adsorption of Ni^{2+} ions. In short, it is concluded that the functional groups of all three types of bananas are rather similar thus it would be difficult to compare and relate the adsorption capacities to the types of banana peels. Perhaps other surface characterization tests would be able to differentiate the types of banana peels using pore characteristics or even the composition of carbon, hydrogen and nitrogen.

4.1.2 CHN Analysis

Using the CHN Analyzer, the composition of Carbon, Hydrogen and Nitrogen for each type of banana peels was determined. The compositions are as shown in the table below:

Table 3: CHN Composition of Banana Peels

	1	2	Avg	1	2	Avg	1	2	Avg
Pisang Berangan	40.34	39.61	39.98	7.49	6.47	6.98	0.43	1.60	1.01
Pisang Emas	39.05	40.08	39.57	5.47	5.63	5.55	1.83	1.92	1.88
Pisang Tanduk	40.63	39.06	39.85	5.73	4.52	5.13	1.89	1.81	1.85

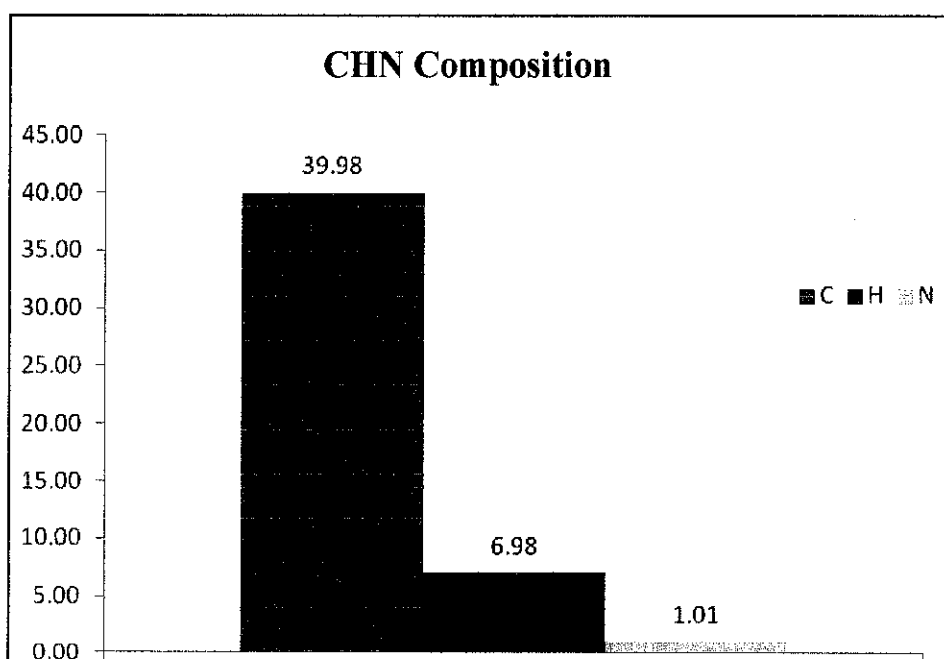


Figure 13: Graph of CHN Composition for Pisang Berangan

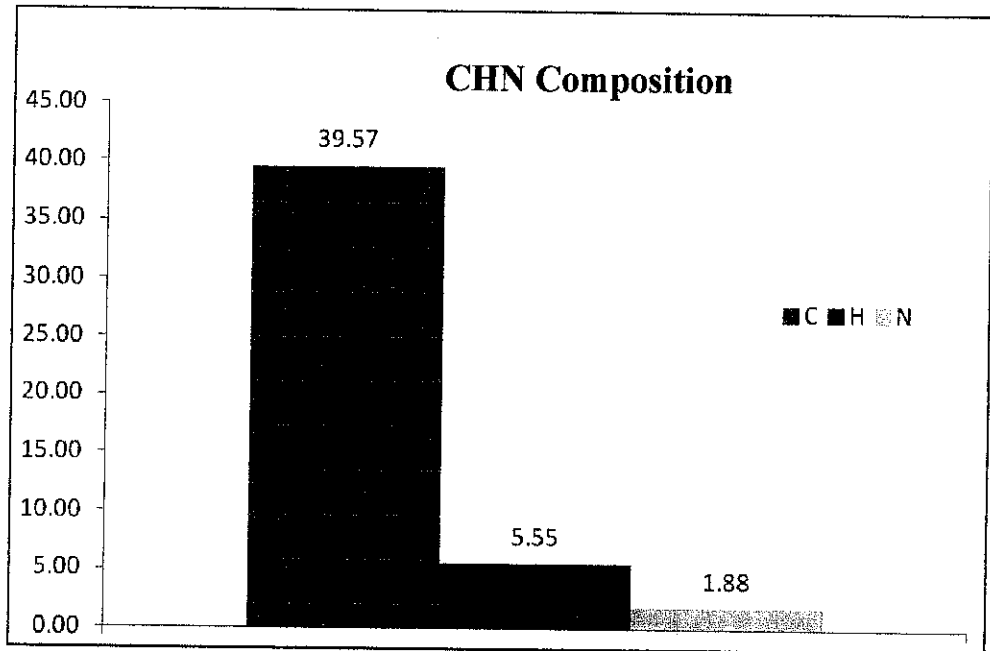


Figure 14: Graph of CHN Composition for Pisang Emas

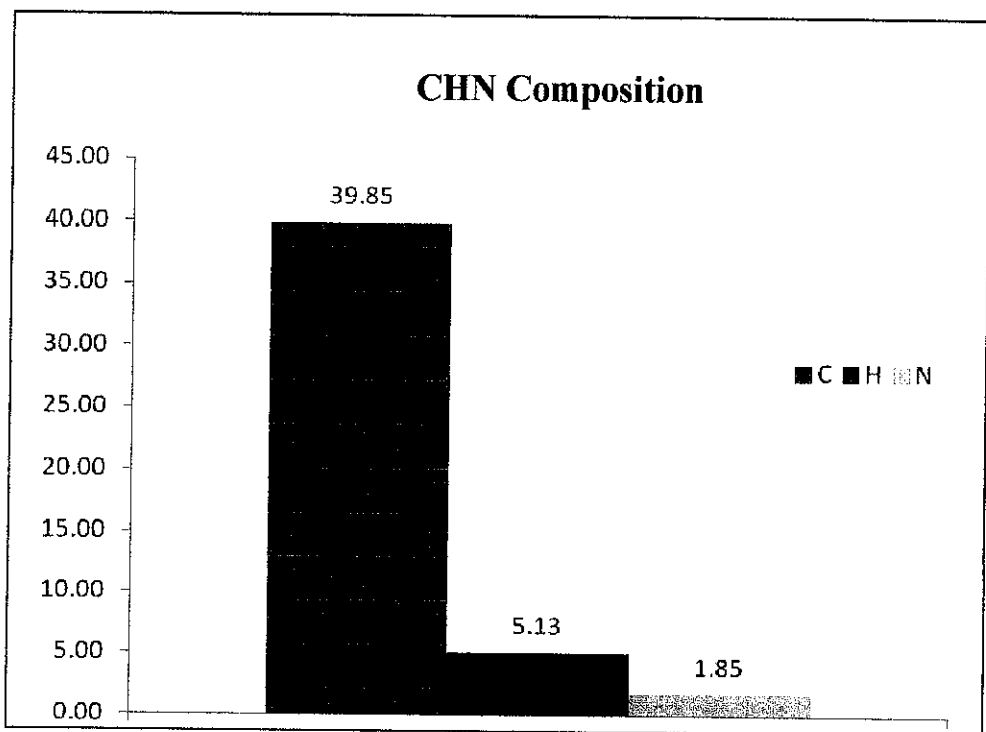


Figure 15: Graph of CHN Composition for Pisang Tanduk

All three types of banana showed almost same average values of Carbon, Hydrogen and Nitrogen. Thus, this is also not able to justify the adsorption capacities of the banana. It is hoped that in the future the Brunauer Teller Method (BET) test will be able to differentiate the banana peels via their pore characteristics.

4.1.3 AAS Analysis

Figure 13 below depicts the absorbance vs. concentration (ppm) of all the prepared solutions (10 ppm, 20 ppm, 30 ppm, 40 ppm and 50 ppm) to ensure that the solutions prepared were accurate for the batch experiments. This graph was generated by the AAS.

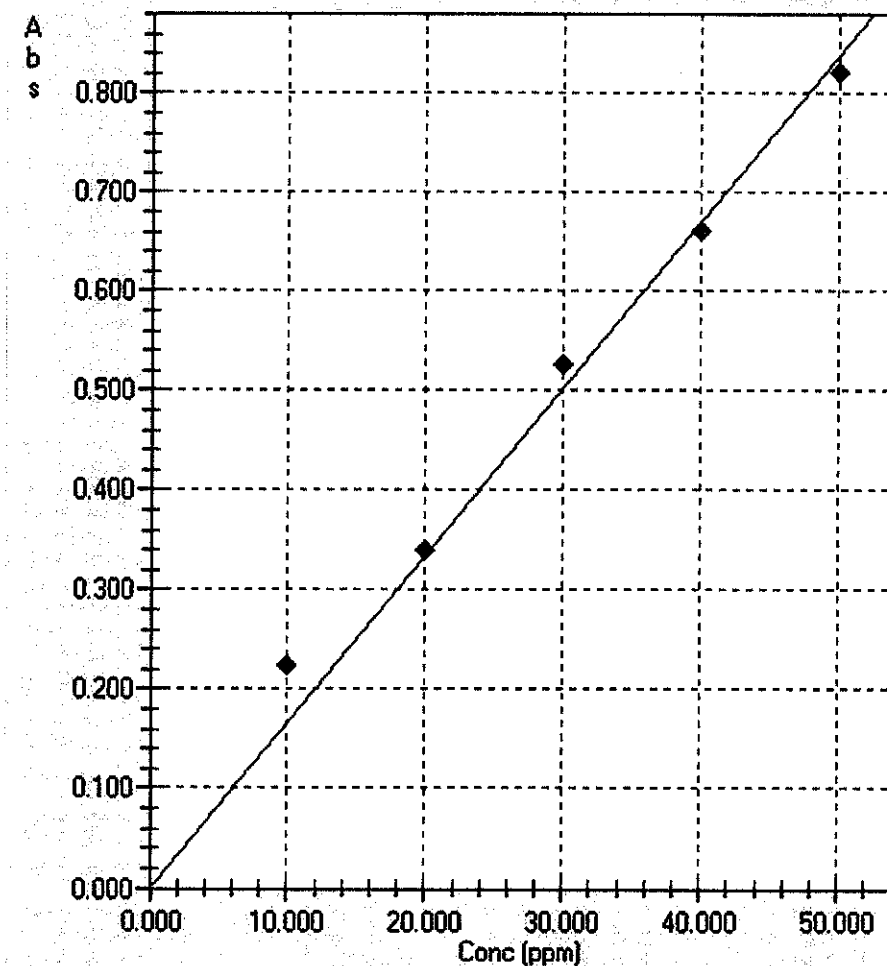


Figure 16: Absorbance vs. Concentration for the prepared solution

- ***Test for Contact Time***

Initial concentration (C_0): 20 ppm

Weight of adsorbent (m): 3g

Volume of solution (V): 200 mL or 0.2 L

Contact time (t): 120 minutes

Table 4: Batch Experiment for Contact Time

0	20.00	20.00	20.00	-	-	-
15	19.42	15.23	16.77	2.90	23.85	16.15
30	19.14	14.39	15.67	4.30	28.05	21.65
45	18.62	14.45	14.13	6.90	27.75	29.35
60	17.14	14.16	13.30	14.13	29.20	33.50
75	15.73	13.87	13.74	21.35	30.65	31.30
90	15.30	13.51	12.42	23.50	32.45	37.90
105	16.91	12.33	10.12	15.45	38.35	49.40
120	13.04	11.90	8.63	34.80	40.05	56.85

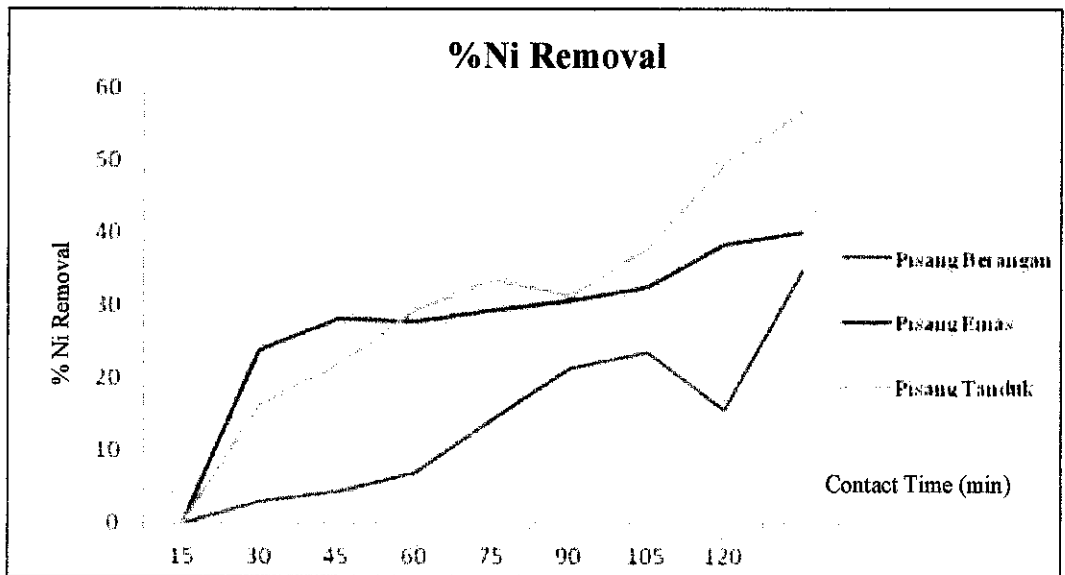


Figure 17: Graph of %Ni removal vs. Contact Time

Figure 17 demonstrates the percentage of Nickel removal versus contact time. The samples were taken at every 15 minutes interval throughout 120 minutes of time. From the graph, Pisang Tanduk starts with a 16.15% Nickel removal and showed a steady increase until the 75th minute. At the 75th minute the graph shows a slight decrease and after that a sharp increase is visible until the 120th minute with a 56.85% of removal. Next, Pisang Emas shows a promising increase at the beginning, it even has a higher removal percentage than Pisang Tanduk. However, starting at the 60th minute, the removal percentage was slightly lower than that of Pisang Tanduk but still higher than Pisang Emas. For Pisang Berangan, it has a significantly low percentage of removal at the beginning with 2.90%, 4.30% and 6.90%. It shows a steady increase until it drops at the 105th minute and experienced a sudden sharp increase until 34.80%. Overall, Pisang Tanduk has the highest

Nickel removal percentage at the 120th minute. It can also be concluded that for this experiment, Pisang Tanduk is the most suitable banana type as an adsorbent which is followed by Pisang Emas and Pisang Berangan. These results confirm the efficiency of banana peels. In the future, perhaps the contact time can be prolonged to find the equilibrium time. In short, when time is varied, Pisang Tanduk is the most suitable banana type with an adsorption capacity of 0.7580mg/g at minute-120 followed by Pisang Emas and Pisang Berangan.

- **Test for Adsorbent Dosage**

Initial concentration (C_0): 20 ppm

Weight of adsorbent (m): 1g, 2g, 3g, 4g, 5g

Volume of solution (V): 200 mL or 0.2 L

Contact time (t): 120 minutes

Table 5: Batch Experiment for Adsorbent Dosage

1	19.61	18.12	19.65	1.95	9.40	1.75
2	16.69	16.45	19.58	16.55	17.75	2.10
3	14.76	14.31	19.47	26.20	28.45	2.65
4	13.04	14.16	18.05	34.80	29.20	9.75
5	10.43	12.42	15.66	47.85	37.90	21.70

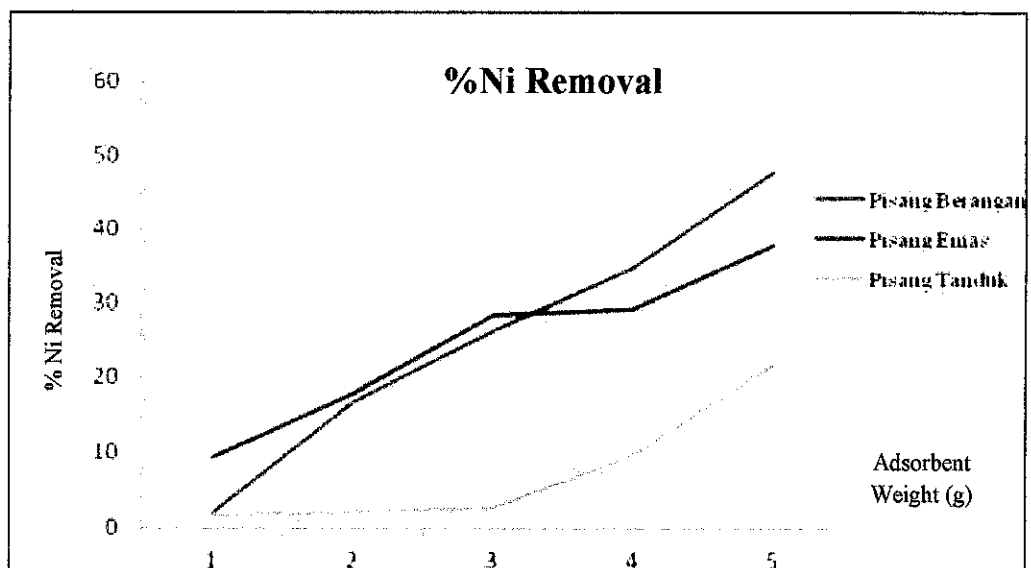


Figure 18: Graph of %Ni removal vs. Weight of Adsorbent

Figure 18 shows the graph of percentage of Nickel removal versus different adsorbent dosage. The weight of the adsorbent was varied from 1g, 2g, 3g, 4g to 5g. Overall, all types of banana peels increases with the increase of dosage. The graph also demonstrates that for this experiment, when we vary the adsorbent dosage, Pisang Berangan is the most suitable type of banana followed by Pisang Emas and Pisang Tanduk. Moreover, Pisang Tanduk experiences a quite slow increase of percentage Nickel removal starting from 1.75%, 2.10% , 2.65% until 9.75%. However, at 5g of adsorbent dosage it suddenly increases to 21.70%. Pisang Berangan had a slow start but then it increased steadily until 47.85%. The optimum adsorbent dosage was not able to be determined however it is evident that the percentage of Nickel removal increases with dosage. For Pisang Emas, it shows a steady increase of the percentage of Nickel removal. With this, it also confirms the efficiency of banana peels an effective adsorbent. Basically, when we vary the adsorbent dosage, Pisang Berangan is the most suitable type of banana with an adsorption capacity of 1.2211mg/g followed by Pisang Emas and Pisang Tanduk.

- ***Test for Different Nickel Concentrations***

Initial concentration (C_0): 10 ppm, 20 ppm, 30 ppm, 40 ppm, 50 ppm

Weight of adsorbent (m): 3g

Volume of solution (V): 200 mL or 0.2 L

Contact time (t): 120 minutes

Table 6: Batch Experiment for Different Nickel Concentrations

10	9.09	9.59	9.58	9.10	4.10	4.20
20	17.04	16.16	15.67	14.80	19.20	21.65
30	23.53	29.10	21.09	21.57	30.60	22.78
40	30.36	20.82	30.89	24.10	27.25	29.70
50	33.79	26.92	32.69	32.42	46.16	34.62

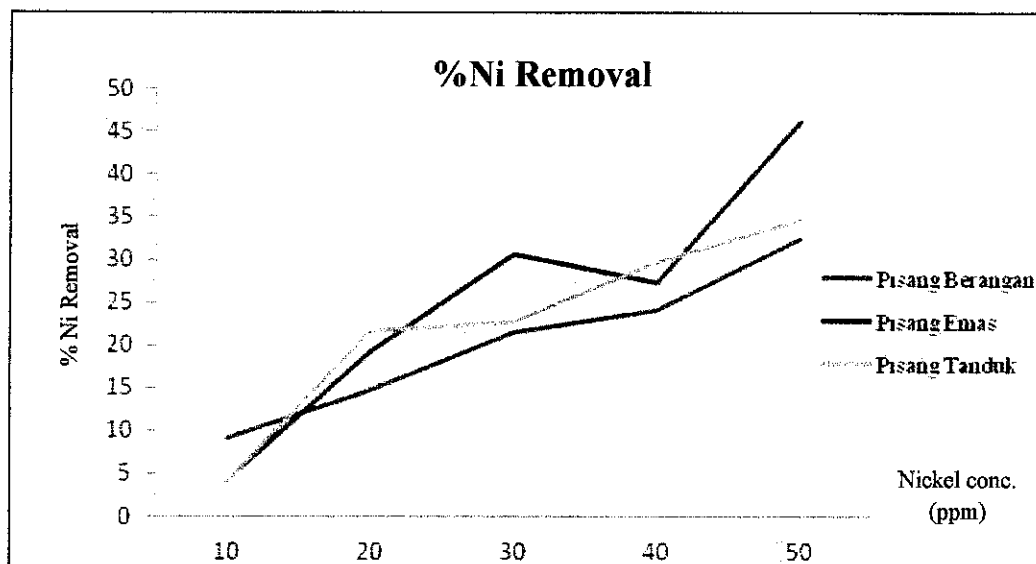


Figure 19: Graph of %Ni removal vs. Nickel Concentrations

Figure 19 depicts the graph of percentage of Nickel removal versus different Nickel concentrations. Nickel concentration was varied from 10ppm, 20ppm, 30ppm, 40ppm to 50ppm. In all, every type of banana peels show an increase in *Ni* adsorption with the increase of Nickel concentration. For Pisang Emas it shows that the adsorption steadily increases until it drops a little at 40ppm and then sharply increases back at 50ppm. Pisang Tanduk increases sharply in the beginning and then steadily increases until 50ppm. Also, Pisang Berangan also increases steadily from 10ppm up until 50ppm. The optimum adsorbent dosage in this experiment was at 50ppm which is the highest concentration as it is also determined that the percentage of *Ni* removal increases with Nickel concentration. With this, it shows that even at a high concentration of 50ppm, all types of banana peels are still able to function as an effective adsorbent. Pisang Emas showed a steady increase and dropped at 40ppm but then experienced a sharp increase until 50ppm and both Pisang Tanduk and Pisang Berangan also showed a steady increased with the increase of Nickel concentration. In the future, even higher Nickel concentrations can be tested to find the optimum Nickel concentration. In short, when we vary the Nickel concentration, Pisang Emas is the most suitable type of banana with an adsorption capacity of 1.5387mg/g followed by Pisang Tanduk and Pisang Berangan.

4.1.3 Isotherm Generation

Freudlich equation,

$$x/m = K_f C_e^{1/n}$$

$$\text{Log}(x/m) = \log K_f + 1/n \log C_e$$

x/m : Mass of adsorbate adsorbed per unit mass of adsorbent, mg adsorbate/g activated carbon

K_f : Freundlich capacity factor, (mg adsorbate/g activated carbon) (L water/mg adsorbate)^{1/n}

C_e : Equilibrium concentration of adsorbate in solution after adsorption, mg/L

$1/n$: Freundlich intensity parameter

Langmuir equation,

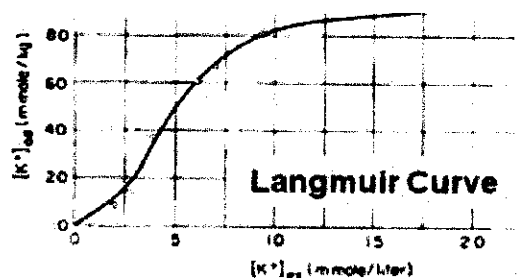
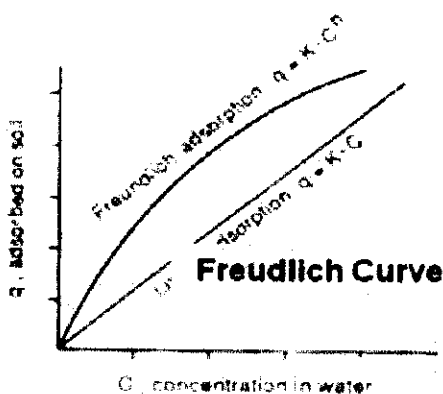
$$x/m = abC_e / (1 + bC_e)$$

x/m : Mass of adsorbate adsorbed per unit mass of adsorbent, mg adsorbate/g banana peels

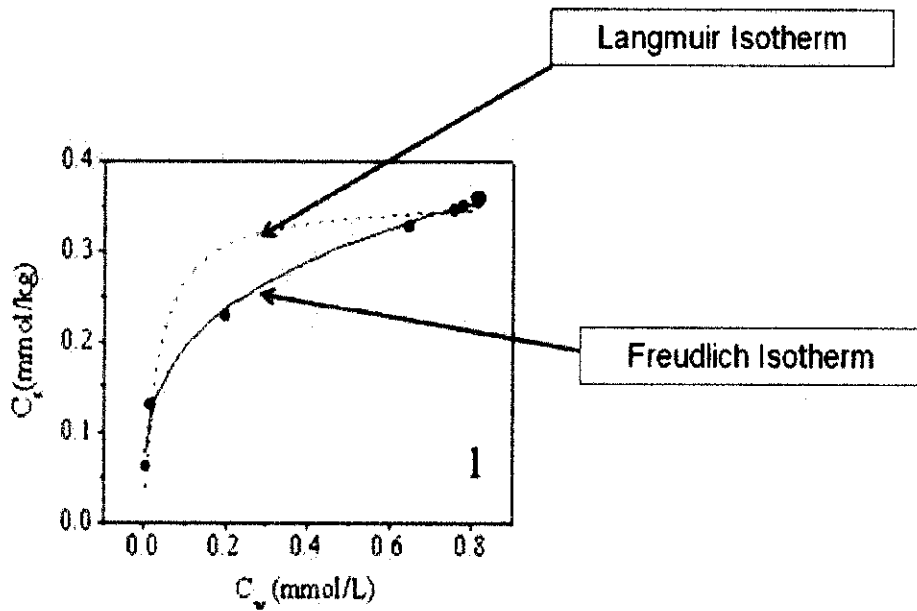
a, b : Empirical constant

C_e : Equilibrium concentration of adsorbate in solution after adsorption, mg/L

Using data of each of the banana peels' type from the third batch experiment (Test for different Nickel concentrations), the *Freudlich* and the *Langmuir* Isotherm was generated. The *Freudlich* and *Langmuir* curve generated will conform to the general curves as shown below to prove that the banana peels' results fit into the isotherms.



For example,



Hence, we can say that the data in the graph plotted is a best fit for Freundlich isotherm rather than Langmuir isotherm.

Table 7: Freundlich and Langmuir Isotherm Data for Pisang Berangan

30	23.53	21.57	0.03595	0.04249	27.81641	-1.37162	-1.44430
40	30.36	24.10	0.04017	0.03294	24.89419	-1.48230	-1.39609
50	33.79	32.42	0.05403	0.02959	18.50824	-1.52878	-1.26737

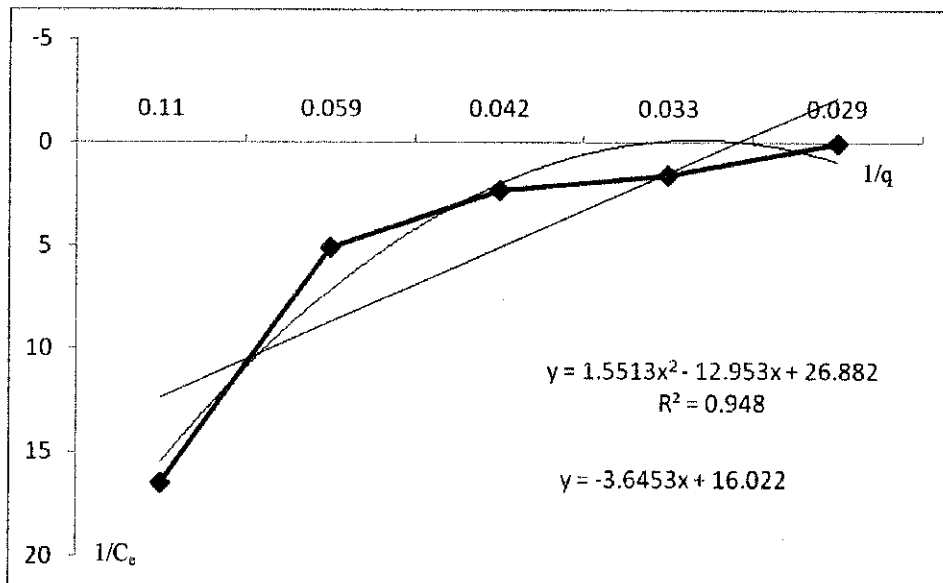


Figure 20: Langmuir Isotherm for Pisang Berangan

Using $y = mx + c$, we know that

$$a/ab = m \text{ and } a/b = c$$

$$y = -3.6453x + 16.022$$

$$1/ab = -3.6453$$

$$1/b = 16.022$$

$$b = 0.062414$$

$$a = -4.39526$$

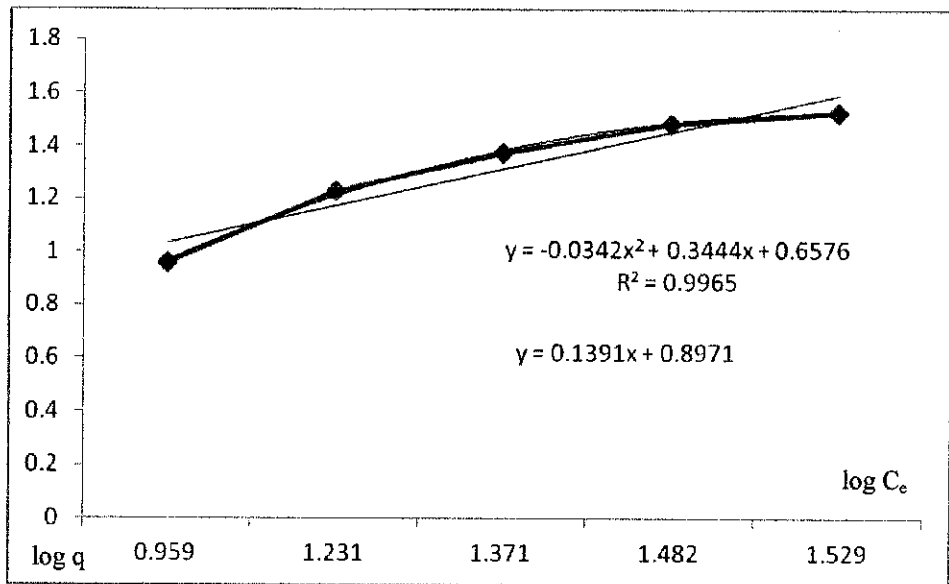


Figure 21: Freundlich Isotherm for Pisang Berangan

Table 8: Freundlich and Langmuir Isotherm Data for Pisang Emas

30	29.10	30.60	0.05100	0.03436	19.60784	-1.463896	-1.29243
40	20.82	27.25	0.04542	0.04803	22.01673	-1.318478	-1.34275
50	26.92	46.16	0.07693	0.03715	12.99883	-1.430076	-1.11390

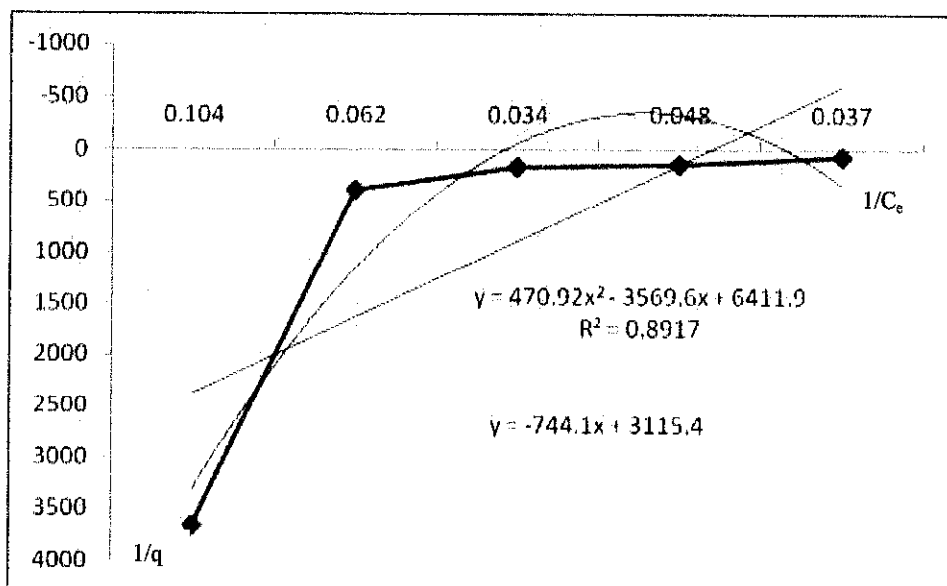


Figure 22: Langmuir Isotherm for Pisang Emas

Using $y = mx + c$, we know that

$$a/ab = m \text{ and } a/b = c$$

$$y = -744.1x + 3115.4$$

$$1/ab = -744.1$$

$$1/b = 3115.4$$

$$b = 3.2098 \times 10^{-4}$$

$$a = -4.18688$$

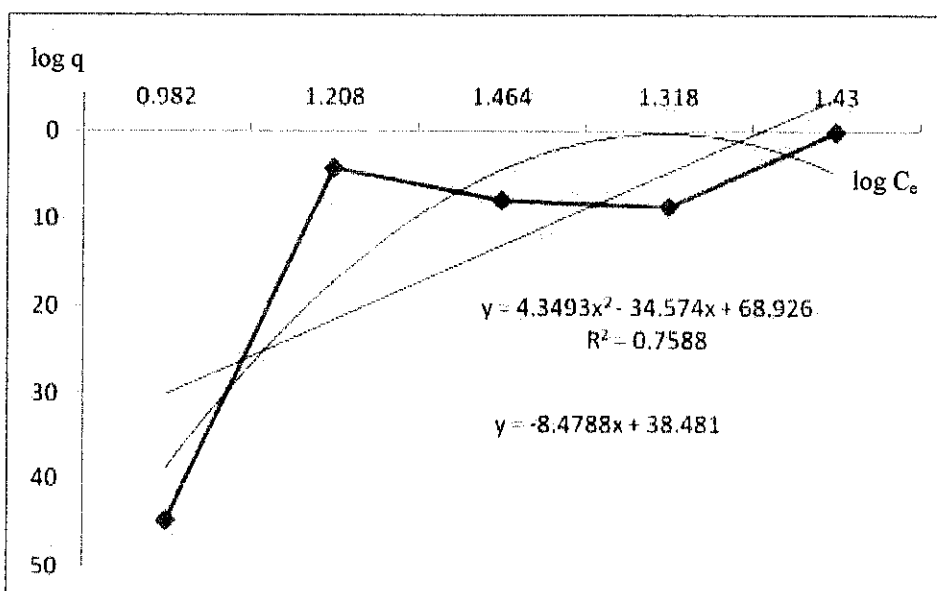


Figure 23: Freundlich Isotherm for Pisang Emas

Table 9: Freundlich and Langmuir Isotherm Data for Pisang Tanduk

30	21.09	22.78	0.03797	0.04742	26.33658	-1.32408	-1.42056
40	30.89	29.70	0.04950	0.03237	20.20202	-1.48982	-1.30539
50	32.69	34.62	0.05770	0.03059	17.33102	-1.51442	-1.23882

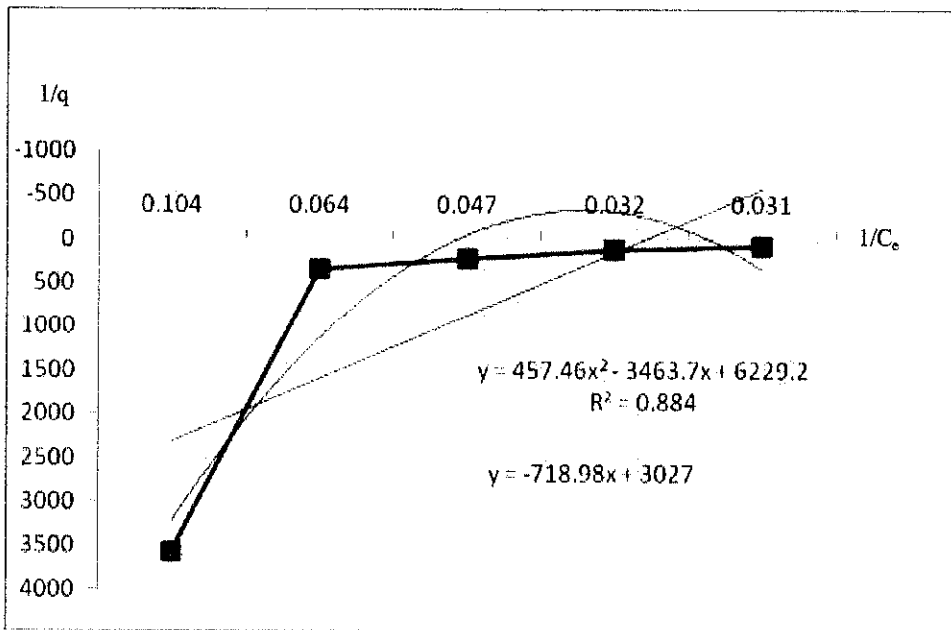


Figure 24: Langmuir Isotherm for Pisang Tanduk

Using $y = mx + c$, we know that

$$a/ab = m \text{ and } a/b = c$$

$$y = -718.98x + 3027$$

$$1/ab = -718.98$$

$$1/b = 3027$$

$$b = 3.3036 \times 10^{-4}$$

$$a = -4.21013$$

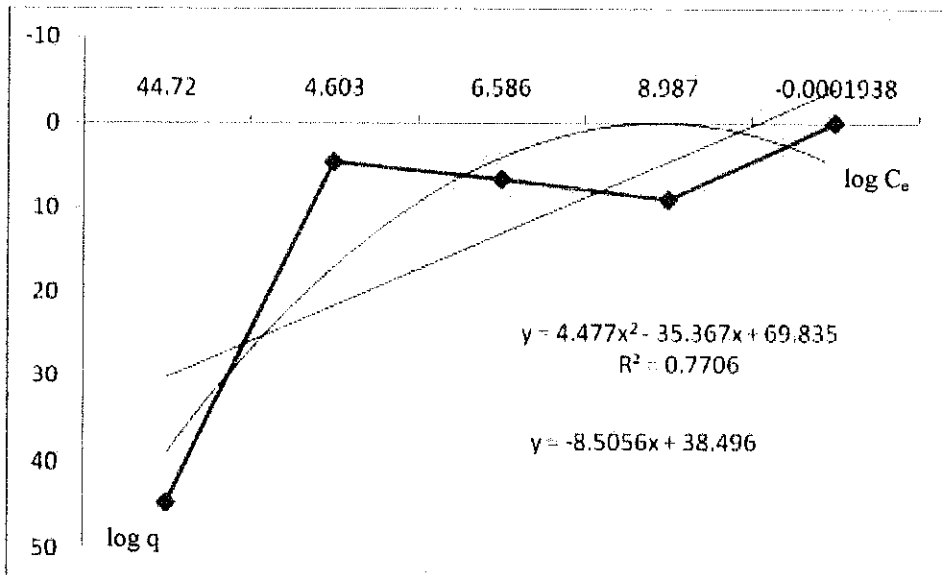


Figure 25: Freudlich Isotherm for Pisang Tanduk

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

There are several conclusions that can be drawn from this research, particularly from the batch adsorption experiments from the objectives that were targeted:

- The banana peels are capable to be an alternative adsorbent in adsorption of nickel as has high surface area, microphous structure and high degree of surface reactivity and the Nickel adsorption activities are well described in the Freudlich and Langmuir isotherm.
- The adsorption of Nickel is increased as the contact time was increased for 120 minutes with 15 minutes interval and the optimum time to reach equilibrium is 120 minute and that for the experiment of Nickel removal versus time, Pisang Tanduk is the most suitable banana type as an adsorbent followed by Pisang Emas and Pisang Berangan. These results confirm the efficiency of banana peels. Also, Pisang Tanduk is the most suitable banana type with an adsorption capacity of 0.7580mg/g at the 120th minute.
- The adsorption of Nickel increased as the adsorbent dosage was increased from 1g, 2g, 3g, 4g and 5g. The optimum weight of banana peel in this experiment is 5g and that for the experiment of Nickel removal versus adsorbent dosage, Pisang Berangan is the most suitable type of banana with an adsorption capacity of 1.2211mg/g followed by Pisang Emas and Pisang Tanduk.
- The adsorption of Nickel increases as the nickel concentration was increased from 10ppm, 20ppm, 30ppm, 40ppm to 50ppm. The optimum Nickel concentration for this experiment is 50ppm. When Nickel concentration varies, Pisang Emas is the most suitable type of banana with an adsorption capacity of 1.5387mg/g followed by Pisang Tanduk and Pisang Berangan.
- This project of study is important for the advancement in the studies of finding alternative methods to remove heavy metal contaminants from wastewater via agricultural adsorbents and finding the best type of banana peel would benefit future studies in determining other variables such as temperature, pH or others.

5.2 Recommendations

- In the future, *Brunauer Emmet Teller Method* can be used to find the relation between adsorption capacity and surface area properties.
- Also, other parts of banana such as the leaves, trunks, stem and etc can be used for adsorption purposes.
- Heavy metals other than Nickel can also be experimented to study the effectiveness of adsorption activities, preferably those that have yet to be tested so far.
- Other variables such as pH and temperature can also be studied in adsorption.
- For future studies, other fruit peels like lemon or orange peels can also be tested.
- Finally, water samples from industrial streams nearby the institution can be obtained in order to study the contents of heavy metal ions in order to target relevant specific heavy metals for adsorption purposes.

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APPENDICES

APPENDIX A

<i>Functional Group</i>	<i>Characteristic Absorption(s)(cm⁻¹)</i>	<i>Notes</i>
Alkyl C-H Stretch	2950 - 2850 (m or s)	Alkane C-H bonds are fairly ubiquitous and therefore usually less useful in determining structure.
Alkenyl C-H Stretch Alkenyl C=C Stretch	3100 - 3010 (m) 1680 - 1620 (v)	Absorption peaks above 3000 cm ⁻¹ are frequently diagnostic of unsaturation
Alkynyl C-H Stretch Alkynyl C≡C Stretch	~3300 (s) 2260 - 2100 (v)	-
Aromatic C-H Stretch Aromatic C-H Bending Aromatic C=C Bending	~3030 (v) 860 - 680 (s) 1700 - 1500 (m,m)	-
Alcohol/Phenol O-H Stretch	3550 - 3200 (broad, s)	-
Carboxylic Acid O-H Stretch	3000 - 2500 (broad, v)	-
Amine N-H Stretch	3500 - 3300 (m)	Primary amines produce two N-H stretch absorptions, secondary amides only one, and tertiary none.
Aldehyde C=O Stretch Ketone C=O Stretch Ester C=O Stretch Carboxylic Acid C=O Stretch Amide C=O Stretch	1740 - 1690 (s) 1750 - 1680 (s) 1750 - 1735 (s) 1780 - 1710 (s) 1690 - 1630 (s)	The carbonyl stretching absorption is one of the strongest IR absorptions, and is very useful in structure determination as one can determine both the number of carbonyl groups (assuming peaks do not overlap) but also an estimation of which types.
Amide N-H Stretch	3700 - 3500 (m)	As with amines, an amide produces zero to two N-H absorptions depending on its type.

APPENDIX B

Calculation for Percent Metal Uptake

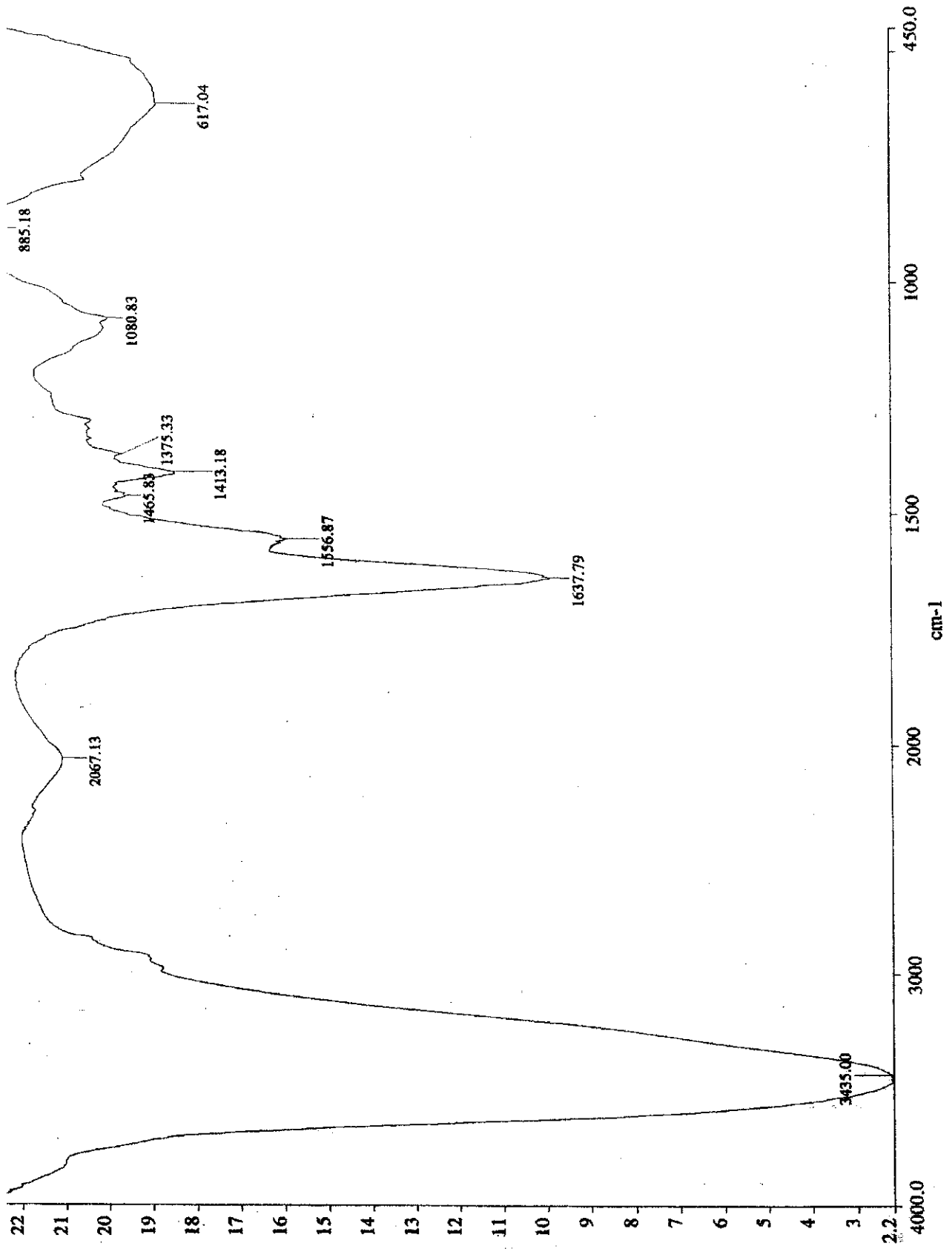
The calculation for metal uptake is such as below:

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

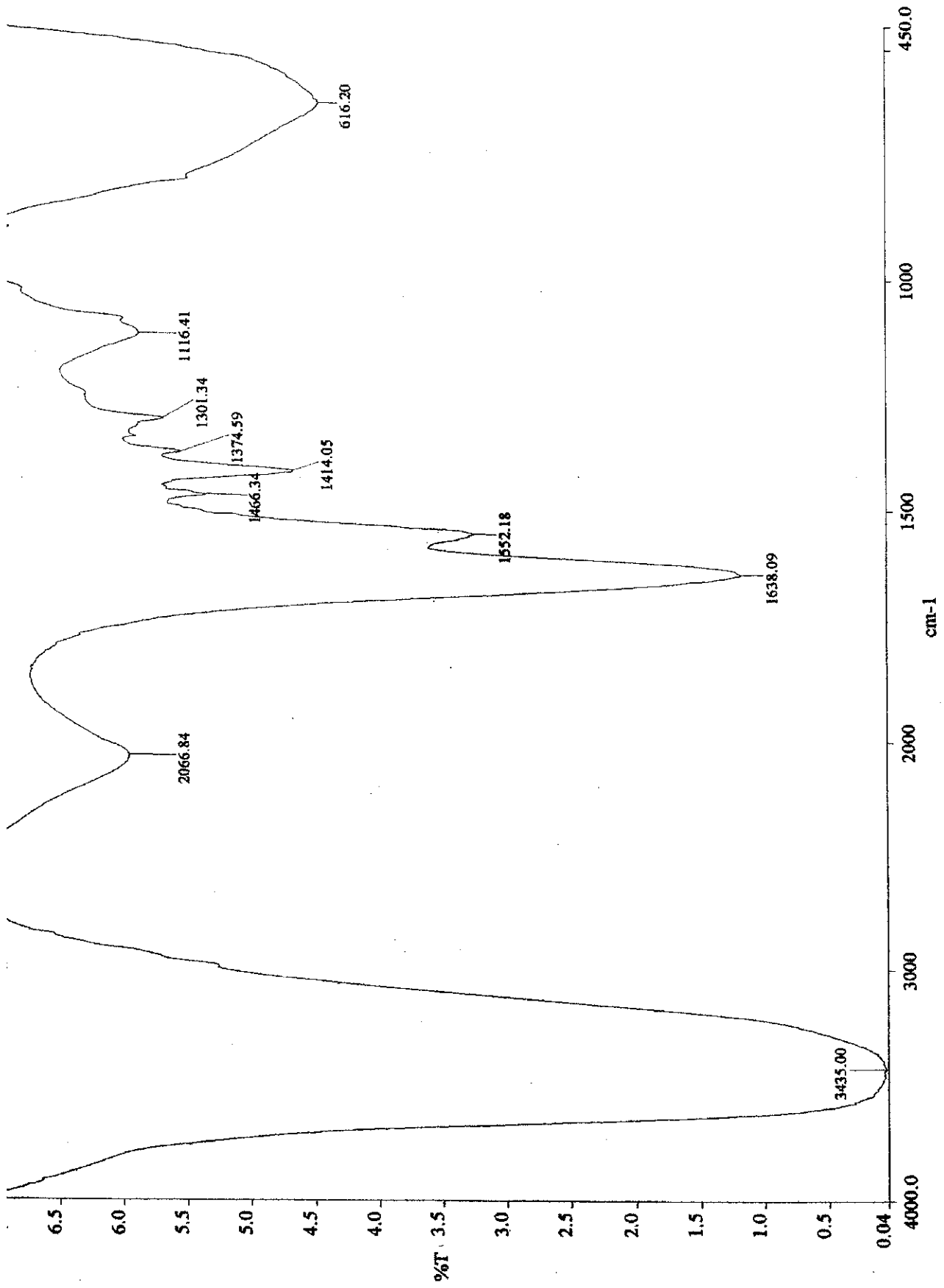
Calculation for Adsorption Capacity

Calculation for adsorption capacity is such as below:

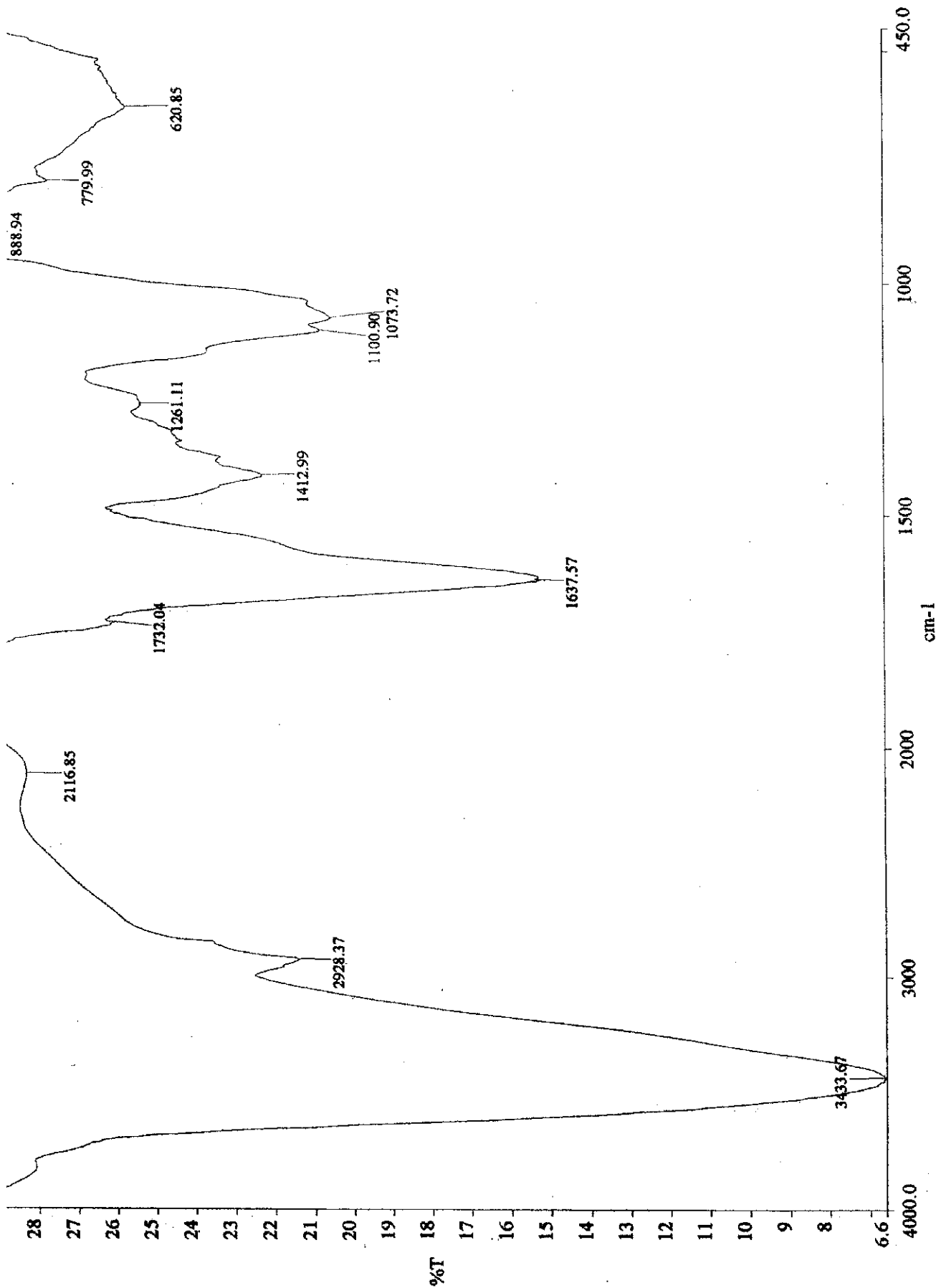
$$\frac{\text{Initial concentration} - \text{Final concentration}}{\text{Weight of banana peels in gram (g)}} \times \text{Volume of solution in litre (L)}$$



c:\pel_data\spectra\pising berangan.002



c:\pel_data\spectra\piscang\banduk.002



c:\pel_data\spectra\pisanang emas.002