Adsorption of Ni (II) from Aqueous Solution in Fixed-Bed Column using Rice Husk-Based Activated Carbon

by

Khadijatul – Akmal Binti Ahmad Hamizi

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

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CERTIFICATION OF APPROVAL

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Approved by,

____

(Dr. Anis Suhaila)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2012

CERTIFICATION OF ORIGINALITY

This is 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.

KHADIJATUL – AKMAL BINTI AHMAD HAMIZI

ABSTRACT

Heavy metal that discharged from industry can affect the health and causing environmental pollution. A variety of wastewater treatment process which have commercially been used including chemical oxidation, ion exchange, reverse osmosis and carbon adsorption. But these processes normally required high cost of implementation and maintenance. Furthermore, production and generation of superior activated carbon that used as adsorbent to remove heavy metal ions from effluent is also expensive. The objective of this project is to obtain and characterize a cheap alternative precursor rice husk-based activated carbon (RHAC) as well as the investigation of its efficiency for removal of Ni (II) ions from aqueous solution using fixed-bed adsorption column. Rice husk, an agricultural waste, is a good alternative source for cheap precursor of activated carbon due to its abundance and constant availability. In this project, rice husk-based activated carbon was prepared via chemical treatment using NaOH as an activation agent prior the carbonization process. Three samples, i.e. raw rice husk (RRH), treated rice husk (TRH) and rice husk-based activated carbon carbonized at 500°C (RHAC) were analyzed for characterization study using FESEM, XRD, FTIR, TGA and BET. The adsorption potential of rice husk- based activated carbon (RHAC) to remove Cu (II) from aqueous solution is investigated using fixed-bed adsorption column. The results obtained from characterization study indicate that carbonization helps to produce higher carbonaceous material in RHAC leading to the formation of high porous structuree and shows that RHAC has a potential to adsorbent for removal of heavy metals. The results obtained from adsorption studies shows that the RHAC has potential to acts as adsorbent for Ni (II) removal.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Heavy metals made up of a group of minerals are among the most important sorts of contaminant in the environment (Wilson, 2011). Heavy metal ores likes iron, chromium, lead and zinc occur as natural elements of the earth crust and are remain as environmental contaminants since they cannot be degraded or destroyed (Duruibe et al., 2007). This heavy metal that causing environmental pollution and can harm living thing has become a critical issue due to rapid industrialization. The heavy metals cause stomach cramps, skin irritations, muscular stiffness, loss of appetite and nausea to human being and produce bioaccumulation in plant and animal. Thus, heavy metals need to be removed from wastewater streams.

The technologies for the treatment that have been commercially uses to remove the heavy metal ions include membrane filtration, carbon adsorption and reverse osmosis (Taha et al., 2011). Among the wastewater treatments, adsorption by activated carbon treatment process turns out to be the most useful and effective method to remove heavy metal content from sewage (Wu and Chen, 2012). For adsorption technologies, this process occurs when the fluid stream passes through a bed or layer of highly porous material called the adsorbent. Thus, this adsorbent will remove the adsorbate, as the adsorbate is disperse to the surface of the adsorbent and are preserved because of weak attractive forces (Harrison, 2008). Adsorption process occurs on the internal surfaces of the materials as shown in Figure 1.1.

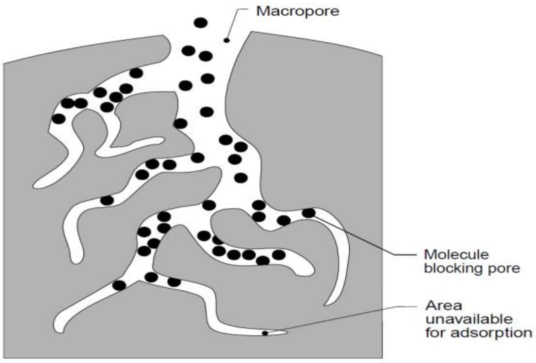


Figure 1.1: Mechanism of adsorption process (Harrison, 2008)

Activated carbon is a microcrystalline with a porous structure can be manufactured by organic material with high carbon content. It has been proved to be the most economical adsorbents for wastewater treatment (Norlia et al., 2011). There are two phases involved in manufacturing activated carbon which are activation and carbonization. As activated carbon can be produced using any organic material with low inorganic components, agricultural waste may have potential to be used as a precursor in producing activated carbon. Producing activated carbon from agricultural waste can convert agricultural waste to a valuable material and reduce the production of activated carbon cost. The most common types of adsorbents used in adsorption technology for wastewater treatment shown in Table 1.1.

Table 1.1: Physical properties of major types of adsorbents (Harrison, 2008)

Adsorbent	**Internal porosity**	**Surface area(m2/gm)**	**Pore volume (cm3/gm)**	**Bulk dry density (gm/cm3)**	**Mean pore diameter (Å)**
Activated carbon	55-75	600-1600	0.80-1.20	0.35-0.50	1500-200
Zeolites	40-55	600-700	0.27-0.38	0.80	300-900
Synthetic polymers	-	1080-1100	0.94-1.16	0.34-0.40	-

As Malaysia has an abundance of agricultural waste, there a lot of precursor used to produce the activated carbon such as coconut husk, rice husk, bamboo, and jackfruit peel (Yee June et al., 2010). This project focuses on producing activated carbon using rice husk as precursor as it is cheaper compared to others because of too much abundance supply. Rice husk is an outermost layer of protection encasing of rice grains and having yellowish color and convex shape. Rice husks are produced by removing the husk form the grain in the first step in the milling process. During milling of paddy about 78 % of weight is received as rice, broken rice and bran and the remaining 22 % of the weight of paddy is known as rice husk (“Rice Husk Ash”, n.d.).

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

i. Excessive heavy metals have been discharged into the environment by several industries due to industrialization and urbanization has created a world-wide environmental problem as they can cause a harmful effect on human physiology. The wastewater treatment processes usually require high operation and maintenance costs.

ii. Commercial activated carbon available in the market is expensive where it is used in wastewater treatment to absorb heavy metal ions from stream.

iii. Rice husk causing some disposal and environmental problems where it generates air pollution and can cause a harmful effect to human and plant.

1.2.2 Significant of the Project

This project aims to obtain and characterize a cheap alternative precursor rice husk-based activated carbon (RHAC) as well as the investigation of its efficiency for removal of Ni (II) from aqueous solution using fixed-bed adsorption column.

1.3 OBJECTIVE

This study embarks on the following objectives:

i. To characterize rice husk-based activated carbon (RHAC).

ii. To investigate adsorption potential of using rice husk-based activated carbon for removal of Ni (II) from aqueous solution using fixed-bed adsorption column.

1.4 SCOPE OF STUDY

This project will focus on producing activated carbon by chemically treat the rice husk with sodium hydroxide and convert it to rice husk-based activated carbon using carbonization process. This project extends the research of adsorption potential for the activated carbon to remove mixed heavy metal from aqueous solution.

1.4.1 Relevancy of the Project

The wastewater treatments which have been used include ion-exchange, membrane processing and others, but adsorption process turns out to be economical alternative method to remove heavy metal content at trace level. But since the cost of implementation for this process is high, producing a low-cost activated carbon has been investigated as economical alternative adsorbent to remove heavy metals from wastewater.

1.4.2 Feasibility of the Project

For this project, the first semester cover the literature review for the current method and types of activated carbon that have been used to adsorb heavy metal ions from aqueous solution. This semester also cover the formulation of methodology that will be used in produce the cheap activated carbon to adsorptive remove heavy metal using rice husk as a precursor. The second semester will cover the experimental work and analyzing data.

CHAPTER 2
LITERATURE REVIEW

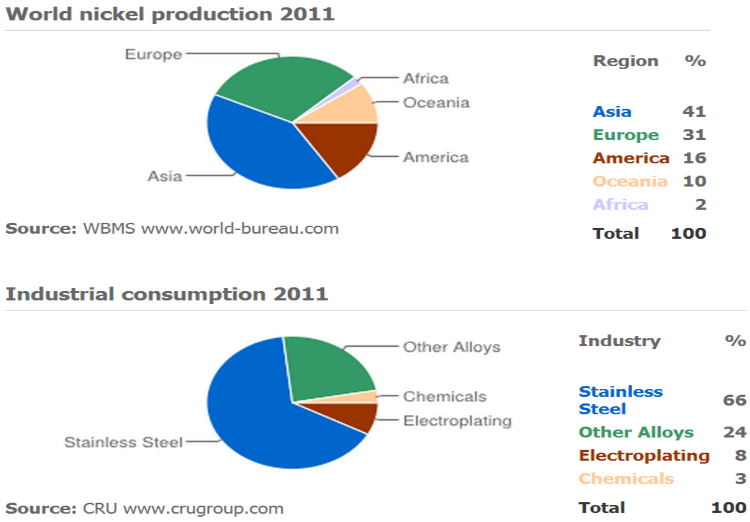
2.1 HEAVY METAL

Heavy metal contamination exists in aqueous waste streams from many industries such as metal plating, mining, tanneries and painting as well as agricultural sources where fertilizer and funicidal spray are intensively used. Cn, Zn, Cr, Ni are examples of harmful wastes produced by industry that cause a risk of contaminating groundwater and other water resources. Heavy metals are not biodegradable and tend to accumulate in living organisms causing various disease and disorder.

Nickel is a metallic chemical element and has been used in alloys for thousands of years. A common use of nickel is in the making of steels. Nickel is also used in production of iron alloys, copper-nickel alloys, and silver-nickel alloys. It is also used in electroplating and in the making of nichrome alloys for resistive heating elements. The world nickel production is still increasing as shown in Figure 2.1 and Table 2.1 which indicates that the amount of nickel ion being discharged from the industries also increases. Although nickel is a nutrient needed in human body, but high dosage will cause health problem.

Table 2.1: Production of primary nickel and nickel usage (International Nickel Study Group, 2011)

SUMMARY	**PRODUCTION OF PRIMARY NICKEL**					
	2005	2006	2007	2008	2009	2010
Africa	**55.5**	**54.5**	**49.1**	**42.1**	**36.4**	**36.0**
America	**307.6**	**313.1**	**317.2**	**304.8**	**239.2**	**229.0**
Asia	**270.6**	**303.5**	**379.4**	**378.6**	**432.3**	**538.0**
Europe	**462.9**	**511.6**	**513.7**	**510.2**	**444.4**	**501.2**
EU27	110.7	116.1	121.8	122.8	81.5	108.3
Oceania	**177.5**	**162.6**	**156.2**	**141.9**	**167.6**	**141.4**
WORLD	**1274.1**	**1345.3**	**1415.6**	**1377.7**	**1319.9**	**1445.6**



World nickel production 2011

Region	%
Asia	41
Europe	31
America	16
Oceania	10
Africa	2
Total	100

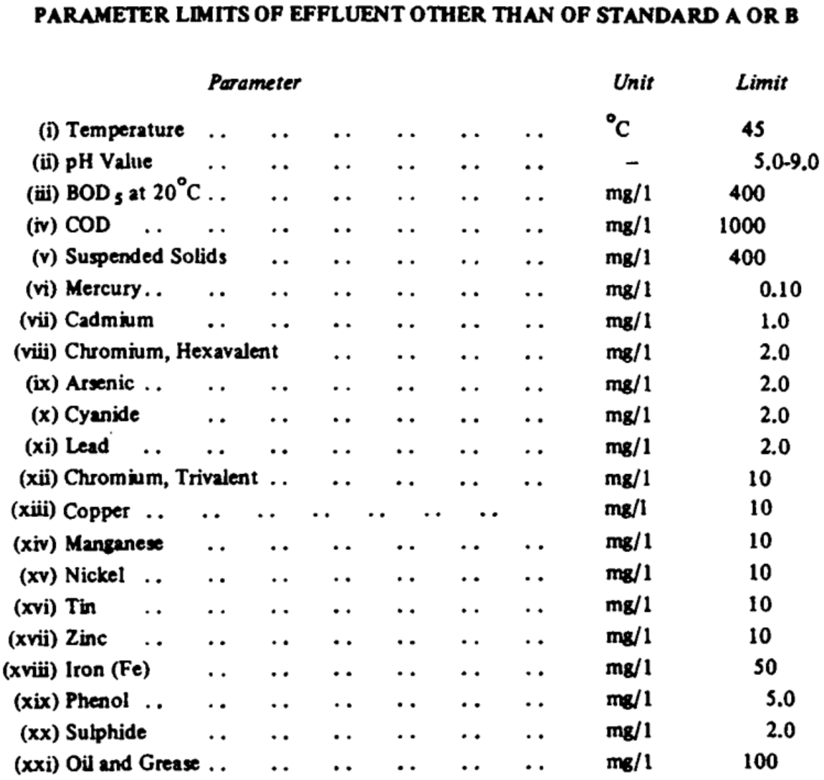
Source: WBMS www.world-bureau.com

Industrial consumption 2011

Industry	%
Stainless Steel	66
Other Alloys	24
Electroplating	8
Chemicals	3
Total	100

Source: CRU www.crugroup.com

Figure 2.1: World nickel production and industrial consumption of nickel (London Metal Exchange, 2012)



PARAMETER LIMITS OF EFFLUENT OTHER THAN OF STANDARD A OR B

Parameter	*Unit*	*Limit*
(i) Temperature	°C	45
(ii) pH Value	–	5.0-9.0
(iii) BOD_5 at 20°C	mg/l	400
(iv) COD	mg/l	1000
(v) Suspended Solids	mg/l	400
(vi) Mercury	mg/l	0.10
(vii) Cadmium	mg/l	1.0
(viii) Chromium, Hexavalent	mg/l	2.0
(ix) Arsenic	mg/l	2.0
(x) Cyanide	mg/l	2.0
(xi) Lead	mg/l	2.0
(xii) Chromium, Trivalent	mg/l	10
(xiii) Copper	mg/l	10
(xiv) Manganese	mg/l	10
(xv) Nickel	mg/l	10
(xvi) Tin	mg/l	10
(xvii) Zinc	mg/l	10
(xviii) Iron (Fe)	mg/l	50
(xix) Phenol	mg/l	5.0
(xx) Sulphide	mg/l	2.0
(xxi) Oil and Grease	mg/l	100

Figure 2.2: Environmental Quality Act 1974, Environmental Quality (Sewage and Industrial Effluents) Regulations 1979

2.2 RICE HUSK

Yee June et al. (2010) said that the utilization of agricultural wastes as precursor activated carbon has attracted the interest of researchers in many years. Many agricultural wastes have been proven to be as a precursor of activated carbon and suitable feedstock in producing activated carbon to remove heavy metal ions from wastewater. One of the agricultural residues that have been given by researches is rice husk.

At about 571 million tonnes rice produced annually, approximately 140 million tonnes of rice husk available for utilization whereby 90% is produced in developing countries (Taha et al., 2011). In year of 2009, Malaysia produces about 2.3 million tonnes of rice per annum in 2 harvests and it is estimated that more than 60% of Malaysian rice husk is simply land filled or burned in-situ resulting environment problems (Nick Zemke, 2009). Utilizing rice husk as a precursor for activated carbon can prevent and eliminate these environment problems. Rice husk prove to be chemically stable and having highly porous structure with high surface area. The ability for rice husk to adsorptive remove heavy metal ions from aqueous solution has been proven in past studies (Taha et al., 2011).

Table 2.2: Comparison of precursor on porous properties

References	Precursor	Pore Volume	BET surface area
Taha et. al (2011)	Rice husk	0.17	253.4
Norlia et al., (2011).	Rambutan seed	0.013327	6.8941

Table 2.3: Typical Composition of rice husk (Chuah et. al., 2005)

Composition	Percent
Cellulose	32.24
Hemicellulose	21.34
Lignin	21.44
Extractive	1.82
Water	8.11
Mineral ash	15.05
SiO_2	96.34
K_2O	2.31
MgO	0.45
CaO	0.41

2.3 ADSORPTION

Commercial technologies for the removal of heavy metal such as chemical precipitation, adsorption, ion exchange and reverse osmosis are often neither effective nor economical (Bhattacharya et. al, 2006). A group of suitable methods exists for the removal of heavy metal from wastewater stream which are precipitation, evaporation, membrane separation. But these methods have several disadvantages which include incomplete metal removal, high reagent and energy requirements. Most of these methods are often ineffective or uneconomical when the heavy metal concentration in the range of 10- 100 ppm and the allowable concentration is less than 1 ppm.

Adsorption process is found to be highly effective, cheap and easy to adapt compare to other treatment processes. Adsorption is well-established and powerful technique for treating domestic and industrial effluents. In wastewater treatment, the most widely used method is adsorption onto the surface of activated carbon. Activated carbon removes heavy metal in wastewater stream. In spite of these characteristics, due to high cost and variable performance of carbon regeneration, single use materials are desirable (Ajmal et. al, 2003). This results lead researchers to search for economic, potential and efficient techniques with low cost of activated carbon used as adsorbent.

2.4 FACTOR AFFECTING ADSORPTION

2.4.1 Particle size

The adsorption capacity of rice husk depends on the surface area available for solute surface interaction, which is accessible to the solute. It is expected that adsorption capacity will increased with a larger surface area which means smaller particle size increases the adsorption capacity. Munaf et. al (1997) said that when the size of rice husk particles increases, metal ions decrease. The smaller adsorption sizes create comparatively larger surface areas and higher adsorption will occur. Tiwari et al. (1995) point out that the adsorption capacity of Hg (II) on rice husk decreased with increasing particle size. Higher adsorption for smaller particle size seems due to larger surface area per unit mass of rice husk. His findings also indicated that in the initial stage of adsorption, Hg (II) adsorption was dominated by diffusion transport.

2.4.2 Carbonization Temperature

The purpose of carbonizing the rice husk is to convert it to activated carbon. Taha et. al. prove that sample carbonized at 800°C showed better evolution and development of pores as compared to those carbonized at 400 and 600°C. The ability to remove metal ion from single metal aqueous solution was found to be improved with the increasing of carbonization temperature.

2.4.3 Contact Time of adsorption activities

The contact time effect of rice husk adsorbent to the adsorption of the metal indicate that as the contact time increase, the metal adsorbed will also increase (Ajmal et. al., 2003). About three hours of adsorption, the rate of adsorption will become constant which means the process has achieved an equilibrium condition (Montanher et. al, 2005). However, the fast kinetics between rice husk and the metal depends on the analytical speed and the removal efficiency. Therefore, this factor need to be considered and must be made constant as the effect of contact time is tested.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

There are two major stages for the overall methodology of this project. The first stage is preparation of rice husk – based activated carbon and second stage is performing experiment to study adsorption and isotherm activity.

STAGE 1

3.1.1 Preparation of raw rice husk (RRH)

i. Rice husk is washed with distilled water several times to remove any adhering or contaminant component.
ii. The rice husk is dried in an oven at 80°C for 24 hours to remove all the volatile components and moisture content exist in the rice husk.
iii. The dried rice husk is grinded by using grinder.
iv. The dried rice husk is sieved to obtain 1mm size of rice husk.

3.1.2 Treatment of raw rice husk (TRH)

1.0M of sodium hydroxide used as activation agent for the rice husk treatment process. The required sodium hydroxide is prepared by dilution of sodium hydroxide using ratio of 1:10 (rice husk : sodium hydroxide).

i. 1.0 M sodium hydroxide is prepared. (refer to APPENDIX I for calculation and preparation)
ii. 80 grams of 1mm size of raw rice husk is digested with 800ml of 1.0M of sodium hydroxide in 1000ml beaker.
iii. Beaker containing sample is sealed using aluminium foil.
iv. Then, sample is stir continuously using magnetic stirrer for 24 hours.
v. After 24 hours, the sample (labelled as treated rice husk) is filtered and washed with distilled water several times until the pH value range of filtrate from 6-7.
vi. The treated rice husk is dried in an oven at 80°C for 24 hours.
vii. Then, treated rice husk (TRH) is weighed.

3.1.3 Preparation of rice husk-based activated carbon (RHAC)

The treated rice husk (TRH) is carbonized to remove non-carbon components (volatile matter contents). Carbonization process is themochemical decomposition of organic material into carbon with the presence of nitrogen flow.

i. 4.5 g of treated rice husk is weighed.

ii. The crucible and crucible lid are wrapped internally using aluminium foil.

iii. After weighed, the treated rice husk is placed into the wrapped crucible and wrapped crucible lid is used to close the crucible.

 *the purpose of wrapping crucible and crucible lid with aluminium foil is to avoid any contact between treated rice husk (TRH) with contaminant component inside the crucible.

iv. The crucible containing rice husk is wrapped externally and is put into the fixed bed activation unit at temperature of 500°C.

v. After carbonizing for 2 hours, fixed bed activation unit is reduced to 25°C.

vi. The sample (labelled as rice husk-based activated carbon) is taken out once the temperature of fixed bed activation unit reached 25°C.

vii. The rice husk-based activated carbon is weighed.

viii. The total yield (Y_t) of rice husk-based activated carbon (RHAC) was calculated as:

$$Y_t = \left(\frac{Weight\ of\ RHAC}{Weight\ of\ TRH}\right) \times 100\%$$

<u>STAGE 2</u>

3.1.4 Fixed bed column study for adsorption of Ni (II)

The purpose of adsorption process is to study the adsorption activities of rice husk-based activated carbon (RHAC) to remove Ni (II) from aqueous solution using fixed-bed adsorption column. The adsorption activities can be observed by using Atomic Adsorption Spectrophotometer (AAS) to determine final concentration of Ni (II) in the filtrate.

i. The fixed bed column is made of clear acrylic tube of 20 mm inner diameter and 300 mm height.

 *The fixed bed column is build from Ion Exchange Unit.

ii. The column performance of Ni (II) adsorption onto RHAC is studied at different inlet Ni (II) concentration (100-500 ppm) and feed flow rate (10-50 ml/min).

   *refer to APPENDIX I for calculation and preparation

iii. The RHAC is placed in fixed bed column with bed depths taken are 80 mm (4.50 g).

iv. The Ni (II) solution is pumped to the column in a down-flow direction.

v. Sample is collected at regular intervals.

vi. The sample is analyzed using AAS to measure the final concentration of Ni (II).

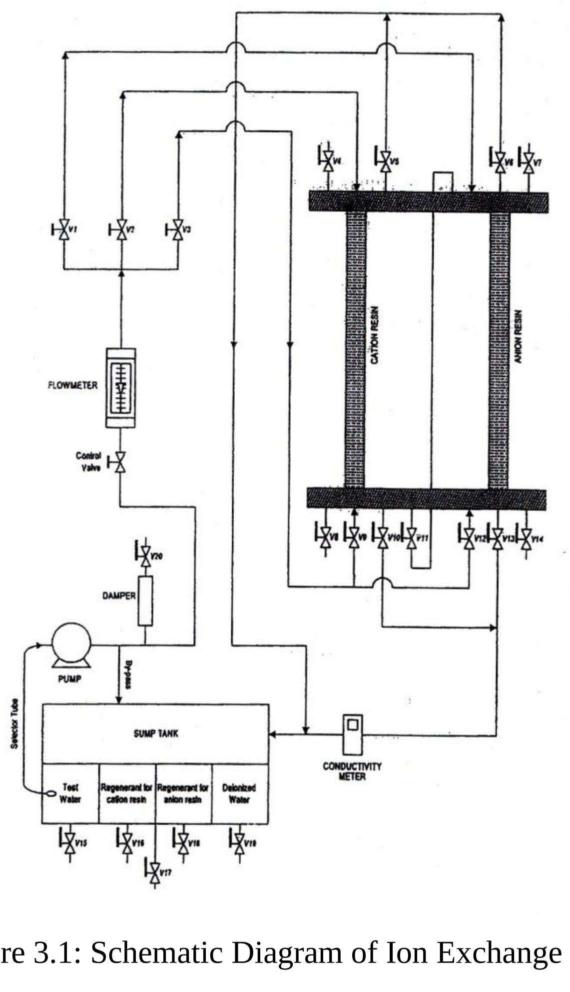


Figure 3.1: Schematic Diagram of Ion Exchange Unit

3.1.5 Column model

The loading behaviour of Ni (II) to be removed from solution in a fixed bed was expressed in term of C_t/C_0 where (C_t = effluent Ni (II) ion concentration and C_0 = influent Ni (II) ion concentration in mg/l). C_t/C_0 is then plotted against time in to obtain breakthrough curve, the maximum column capacity, q_{total} (mg, for a given feed concentration and flow rate is equal to the area under the plot of the adsorbed Ni (II) concentration, C_{ad} ($C_{ad} = C_0 - C$) (mg/l) versus effluent time (t, time) and is calculated from Equation (1):

$$q_{total} = \frac{QA}{1000} = \frac{Q}{1000}\int_{t=0}^{t_{total}} C_{ad}dt \tag{1}$$

Where

t_{total} = total flow time (min)

Q = volumetric flow rate (ml/min)

A = area under the breakthrough curve

The equilibrium uptake (q_{eq}) is calculated as shown from Equation (2):

$$q_{eq} = \frac{q_{total}}{m} \tag{2}$$

Where

m = total dry weight of RHAC in column (g)

The total amount of Ni (II) sent to the column (W_{total}) is calculated as shown from Equation (3):

$$W_{total} = \frac{C_0 Q t_{total}}{m} \tag{3}$$

Total removal percent of Ni (II) is the ratio of the maximum capacity of the column (q_{total}) to the total amount of Ni (II) sent to the column (W_{total}).

$$Y = \frac{q_{total}}{W_{total}} \tag{4}$$

3.1.6 Characterization of RRH, TRH, RHAC, CAC, and adsorption Analysis

Samples before and after adsorption study are analyzed using several equipments as stated in Table 3.2. List of samples for analysis study are shown in Table 3.1.

Table 3.1: List of samples for analysis study

No.	Lab Instrument	Label
1.	Raw Rice Husk	RRH
2.	Treated Rice Husk	TRH
3.	Rice Husk-Based Activated Carbon	RHAC
4.	Commercial Activated Carbon	CAC

Brunauer, Emmett, Teller Surface Area Analyzer (BET)

This equipment is used to study the porous properties of RHAC.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is preferred method of infrared spectroscopy that can identify the functional group of the samples. This equipment measures all the infrared frequencies simultaneously where the amount of material present inside the samples can be determined by the size of the peaks in the spectrum.

X-ray Diffraction (XRD)

XRD is used to investigate the crystalline and amorphous phases of the samples. This analysis is performed with 2θ values ranging from 2° to 80°.

Field Emission Scanning Electron Microscope (FESEM)

FESEM is used to determine silica and oxygen content in the sample. This equipment also used to study the morphological characteristics of the sample as it can visualize very small topographic details on the surface or layer of the sample or fractioned objects.

Thermogravimetric Analyzer (TGA)

TGA measures changes in weight of a sample in relation to changes in temperature in inert or oxidative atmosphere. The measured weight loss curve gives information on changes in composition of sample, thermal stability and kinetic parameters for chemical reactions in the sample.

Atomic Adsorption Spectroscopy (AAS)

Atomic adsorption spectroscopy is an analytical technique which is capable on determining the presence of metals in liquid samples and the concentration of metal ions in the samples by measuring the adsorption of light of free atoms. This project study uses AAS to determine the final concentration of metal ion in the filtrate after adsorption process.

3.2 EQUIPMENTS AND CHEMICALS

Table 3.2: List of lab instruments required

No.	Lab Instrument
1.	BET (Brunauer, Emmett, Teller) Surface Area Analyzer
2.	FTIR (Fourier transform infrared spectroscopy)
3.	XRD (X-ray diffraction)
4.	FESEM (Field emission scanning electron microscope)
5.	TGA (Thermogravimetric analyzer)
6.	AAS (Atomic absorption spectroscopy)
7.	CHNS Element Analyzer
8.	Ion exchange unit
9.	Fixed bed activation unit
10.	Oven
11.	Grinder
12.	Calibrated pH water
13.	Electrical siever

Table 3.3: List of chemicals required

No.	Chemical
1.	Sodium Hydroxide, NaOH
2.	Nickel Nitrate, $Ni(NO_3)_2$
3.	Distilled water

CHAPTER 4

RESULT AND DISCUSSION

4.1 PREPARATION OF RAW RICE HUSK

Preparation of raw rice husk includes the process of cleaning, drying, grinding and sieving the rice husk. The main purpose of this processes are to remove impurities and moisture from rice husk and to obtain 1mm size of rice husk. Based on the observation during cleaning process, a lot of paddy and other impurities were floating on the water. The colour of raw rice husk is dull brown.

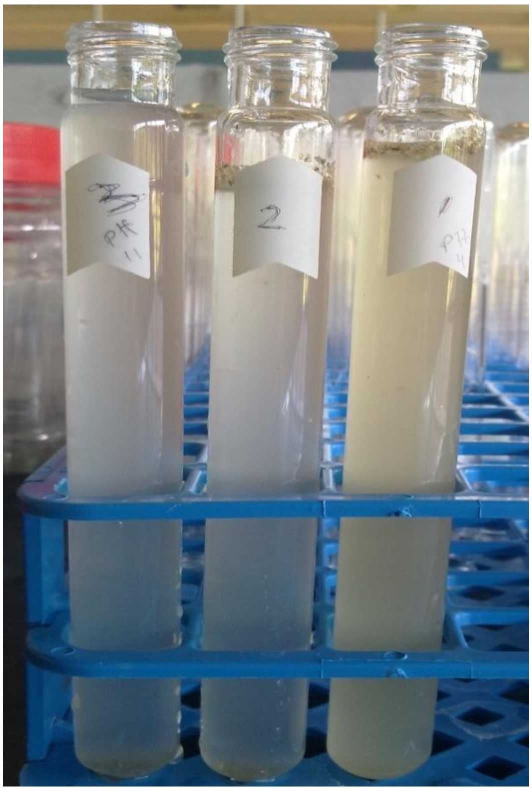


Figure 4.1: Sample of impurities

4.2 TREATMENT OF RAW RICE HUSK WITH SODIUM HYDROXIDE

The major purpose of this process is to remove the silica content from raw rice husk using ratio of 1:10 where 80 gram of raw rice husk is treated with 800 ml of 1.0M sodium hydroxide. This treatment is done for 24 hours with continuous stirring. The reaction involved shown in Figure 4.2 and the observation data of this process shown in Table 4.1.

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$

Figure 4.2: Reaction of silicon oxide with sodium hydroxide

Table 4.1: Observation data

Treatment	Colour	Weight (g)
Before	Dull brown	80
After	Orange	35

Table 4.1 reveals that there is changes in weight of raw rice husk where the yield of treated rice husk (TRH) obtained was found to be 44 %. The reduction in weight was due to the lost of silica content from raw rice husk.

Figure 4.3: The treatment process of raw rice husk

4.3 CARBONIZATION OF TREATED RICE HUSK

The carbonization process was conducted at 500°C with nitrogen flow as inert gas using Fixed Bed Activation Unit. After 4.5 gram of treated rice husk (TRH) was carbonized at 500°C for 2 hours, the colour of TRH sample changes from orange to black. The yield of RHAC obtained was found to be 40%. The reduction in weight after carbonization process was due to the lost of the volatile matters during rapid heating of carbonization process (Rahman et al, 2000). Table 4.2 shows that the weight of the samples was reduced due to the reduction of certain elements. These elements can be determined using FESEM.

Table 4.2 Observation data

Carbonization	Colour	Weight (g)
Before	Orange	4.5
After	Black	1.8

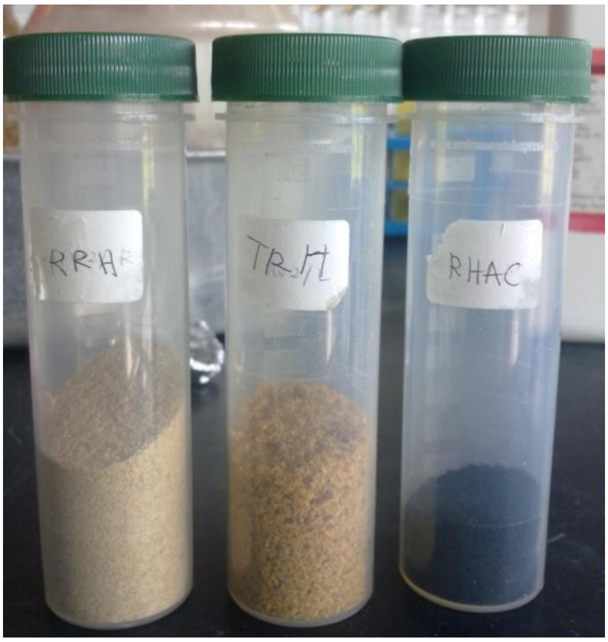


Figure 4.4: Samples of RRH, TRH and RHAC

4.4 CHARACTERISTICS OF RRH, TRH, AND RHAC

4.4.1 Field emission scanning microscope (FESEM)

Two samples were analysed using FESEM/EDX (Energy-dispersive X-ray spectroscopy) to determine elements presence in the samples. Figure 4.5 reveal changes in main component of RRH and RHAC samples. Based on the results shown in Figure 4.5, it can be observed that oxygen content was decreased. Meanwhile, the amount of carbon was found to be increased after raw rice husk was being treated with NaOH and carbonized with nitrogen flow at 500°C for 2 hours. These observations can be explained due to the destruction of hydroxyl groups and carbon—oxygen bonds in cellulose and hemicellulose that occur during carbonization process which lead to the losses of a large number of hydrogen and oxygen which caused the volatile matter to vaporize (Lin et al, 2003). As the volatile matter in rice husk remove during carbonization process leaving non-volatile matter to stay in carbonized rice husk which caused the carbon content becomes constant (Tzong, 2004). Figure 4.5 also shows that the amount of silica weight was decreased. The reduction of silica content could be due to treatment of raw rice husk with NaOH. Silica is the most abundant inorganic element in rice husk where 67.30% of silicon oxide exists in rice husk ash. In this treatment, silica in the ash is extracted with sodium to form sodium silicate (NA_2SO_3). Sodium silicate can be removed easily from rice husk by washing repeatedly with distilled water as it has a highly solubility in water and maintains stability while in neutral or alkaline solutions (Badger, 2012).

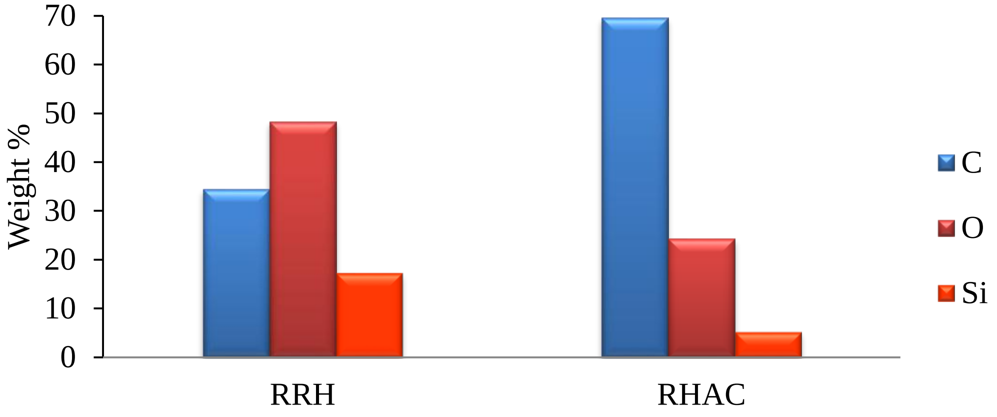


Figure 4.5: Carbon, Oxygen and silica content of RRH and RHAC determined by FESEM/EDX

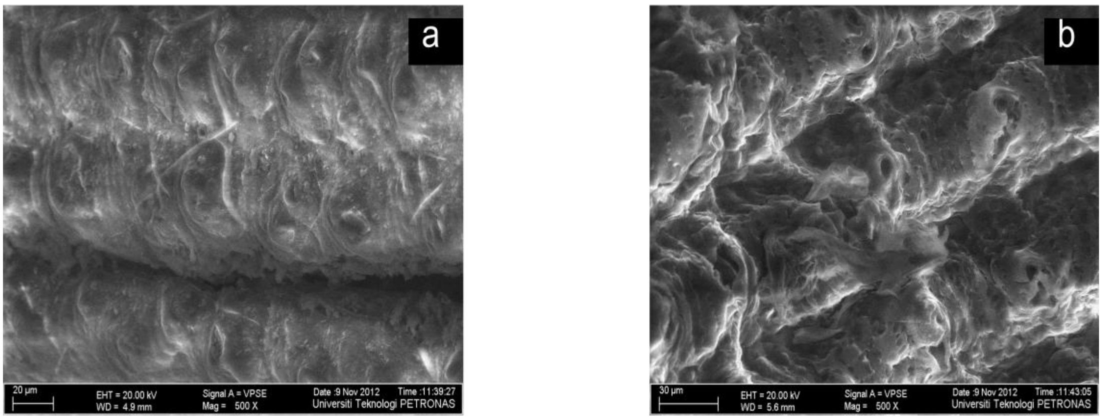


Figure 4.6: Morphology of (a) RRH and (b) RHAC

FESEM studies were carried out for RRH and RHAC samples in order to study the effect of treatment process with NaOH and carbonization process with nitrogen flow at 500°C for 2 hours on porosity development. Morphology of RRH and RHAC can be observed from Figure 4.6(a) and (b) respectively. FESEM micrograph of RRH shows no presence of pores while FESEM micrograph of RHAC shows the presence of many fine pores which result due to treatment process with NaOH to form sodium silicate and carbonization process. Sodium silicate can easily removed by washing process with distilled water causing creation of pores in the interior structure. (Kennedy et al, 2004) points out that dissolution of lignin and other material components from raw rice husk cause the formation of pores on the rice husk outer epidermis. The formation of pores expected to be increased due to removal of volatile matter such as oxygen, hydrogen and nitrogen during carbonization process. Similar studies have been reported by other thesis of preparing activated carbon from rice husk with chemical activation (Daffalla et al, 2011), rice husk by $ZnCI_2$ activation and subsequent gasification with CO_2 (Yahaya et al, 2011), and groundnut shell by $ZnCI_2$ activation (Malik et al, 2006). Many researches shown that these well development pores can contribute to better adsorption capability of activated carbon for removal of heavy metal ions. Figure 4.7 shows similar observation by other researcher in their work of preparing activated carbon from rice husk.

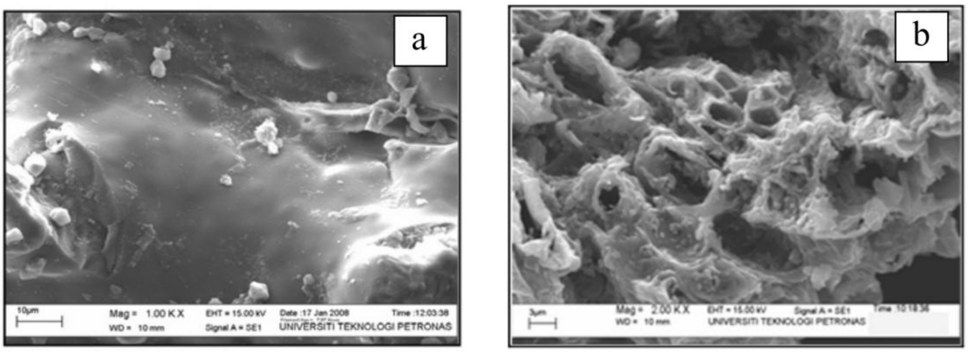


Figure 4.7: Morphology of (a) RRH and (b) RHAC (Taha et al, 2011)

4.4.2 Thermogravimetric analyzer (TGA)

Figure 4.7 shows that TG curves of RRH and TRH exhibit two weight losses steps with temperature in an inert atmosphere. The weight losses over temperature indicate the composition of the sample and thermal stability. Various elements of the samples decompose as the temperature increased. The curves in Figure 4.8 show an initial weight loss below 100°C. These curves might be due to moisture elimination. The second weight loss occurs from 150 to 450°C could be due to decomposition of three major components which are cellulose, hemicelluloses, and lignin. These observations approved with the reports of (Shafizadeh & DeGroot, 1976) and (Antal, 1983). These reports stated that three major constituents of cellulose, hemicellulose, and lignin in the lignocellulose materials are chemically active and decompose thermochemically between 150 and 500°C; hemicellulose mainly between 150 and 350°C, cellulose between 275 and 350°C and lignin between 250 and 500°C. At 800°C, the ash content of TRH is lower than RRH as rice husk has high ash content varying 18 to 20% which mainly consists 80 to 90% of silica. This reduction ash content could be due to leaching of silica from RRH during treatment process with NaOH. Suppose TGA analysis can determine ash content of rice husk sample by straight line toward the end of TG curves, but it seems that testing time is not sufficient to determine the ash content shown in Figure 4.12, 4.14, and 4.16.

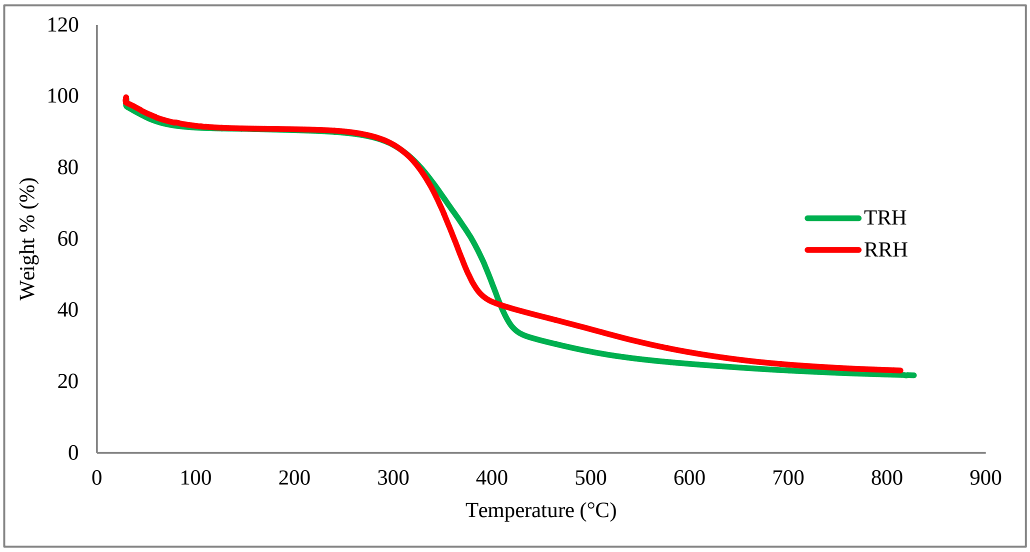


Figure 4.8: Thermal analysis of RRH and TRH (temperature)

Similar observations have been reported by other researchers in their work of preparing activated carbon from rice husk (Angelova et al, 2010) and durian shell (Yee Jun et al, 2010) as shown in Figure 4.9 and 4.10 where the curve shows that the first stage range from 59 to 200°C and presents a 4.5 % weight loss due to moisture release from the material. Between 200 to around 300°C the mass loss is as high as 14%. In the temperature range of 300 to 400°C, a weight of 46% of weight loss was observed. This weight loss is attributed to the decomposition or oxidation of organic compounds such as lignin and cellulose in the raw durian shell.

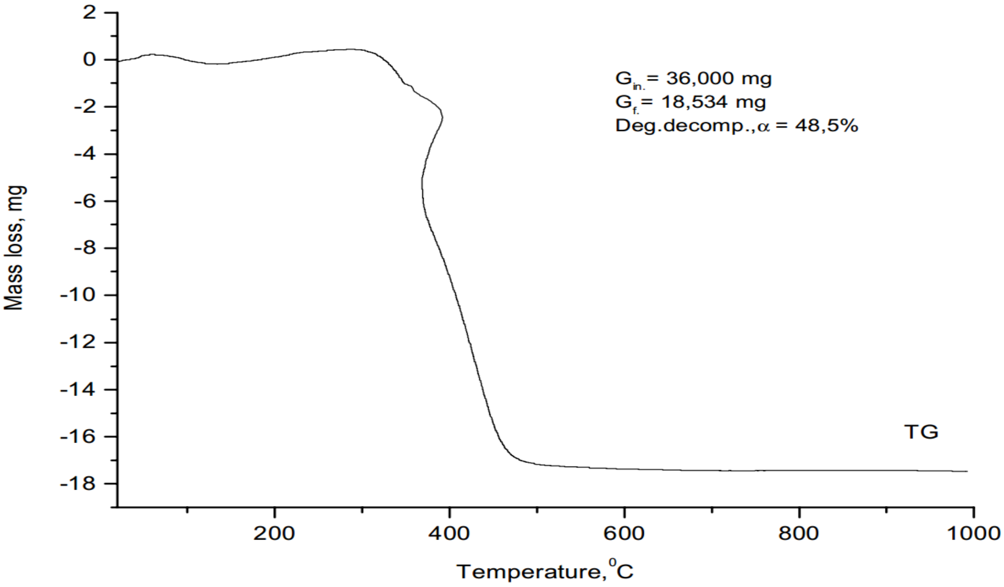


Figure 4.9: TGA curves of rice husk (temperature) (Angelova et al, 2010)

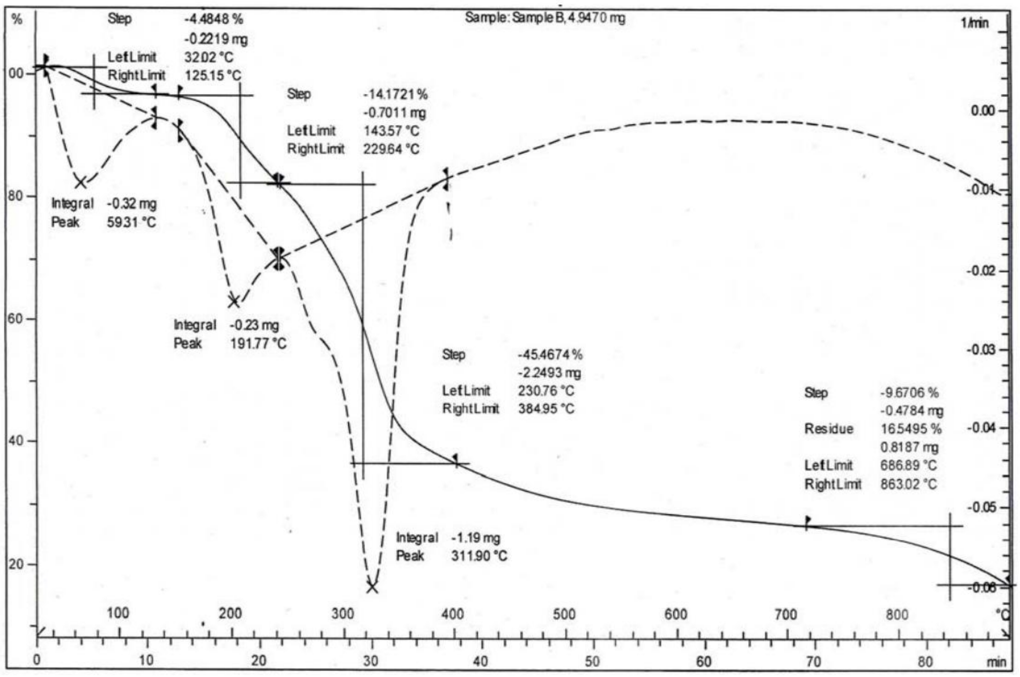


Figure 4.10: TGA curves of raw durian shell (temperature) (Yee Jun et al, 2010)

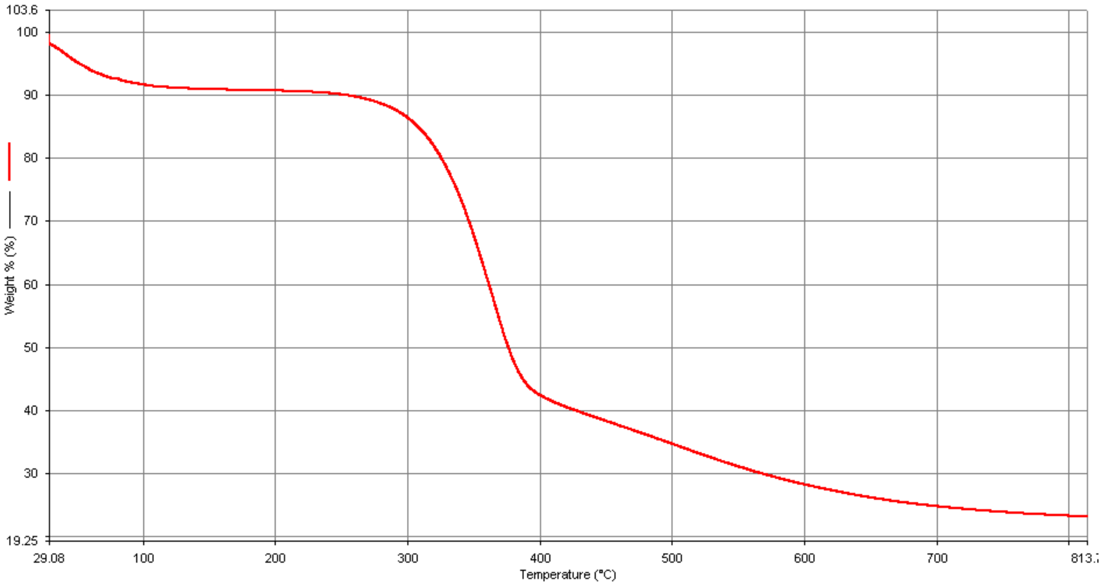


Figure 4.11: Thermal analysis of RRH (temperature)

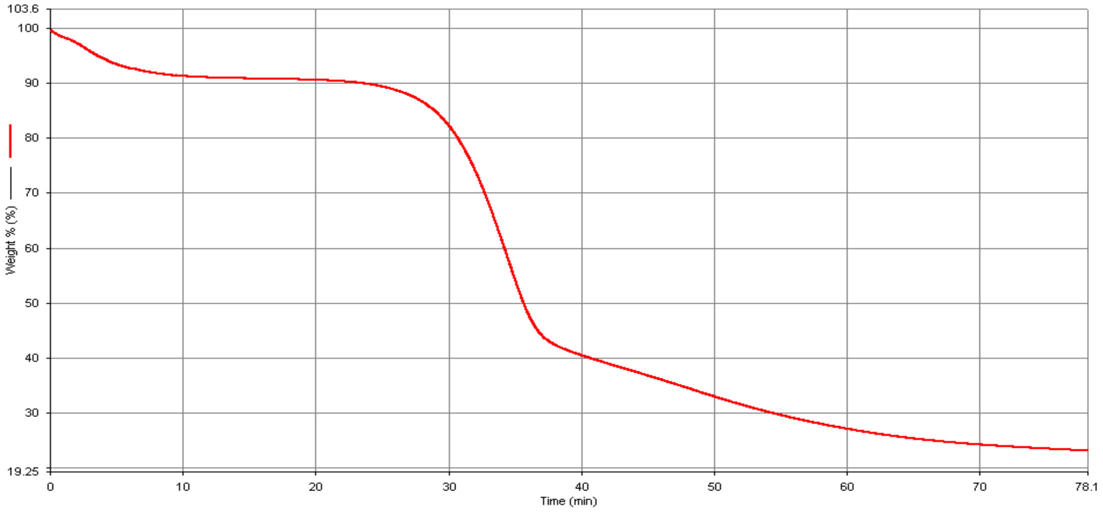


Figure 4.12: Thermal analysis of RRH (time)

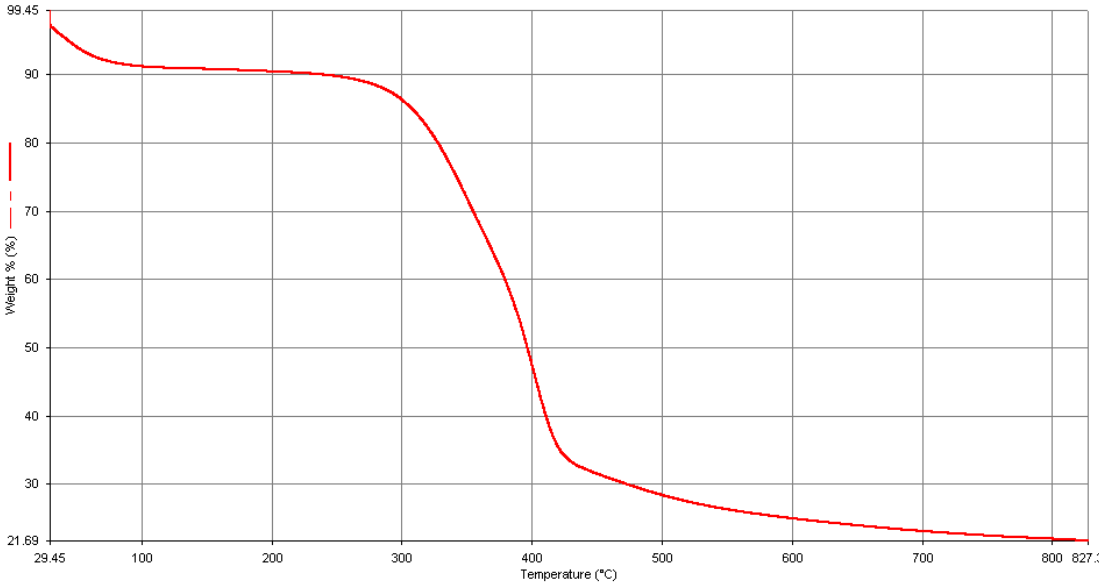


Figure 4.13: Thermal analysis of TRH (temperature)

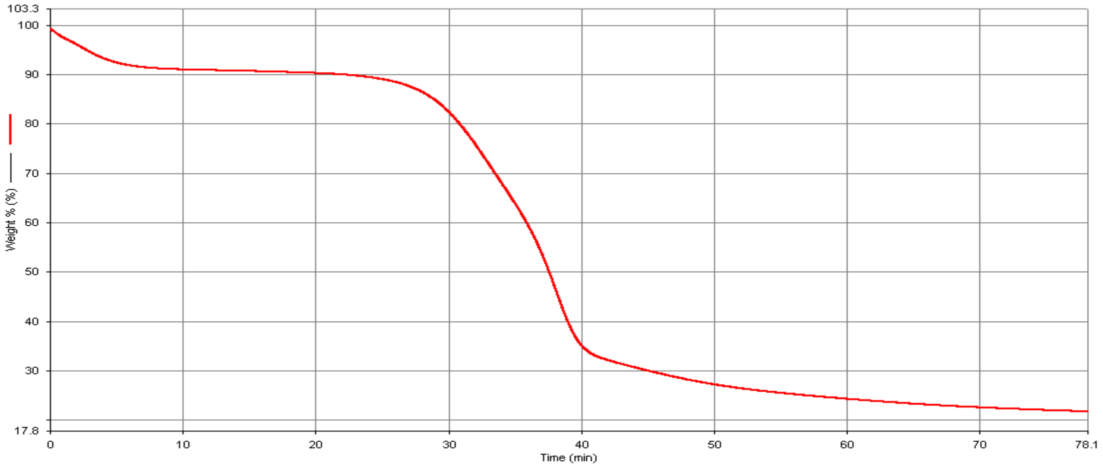


Figure 4.14: Thermal analysis of TRH (time)

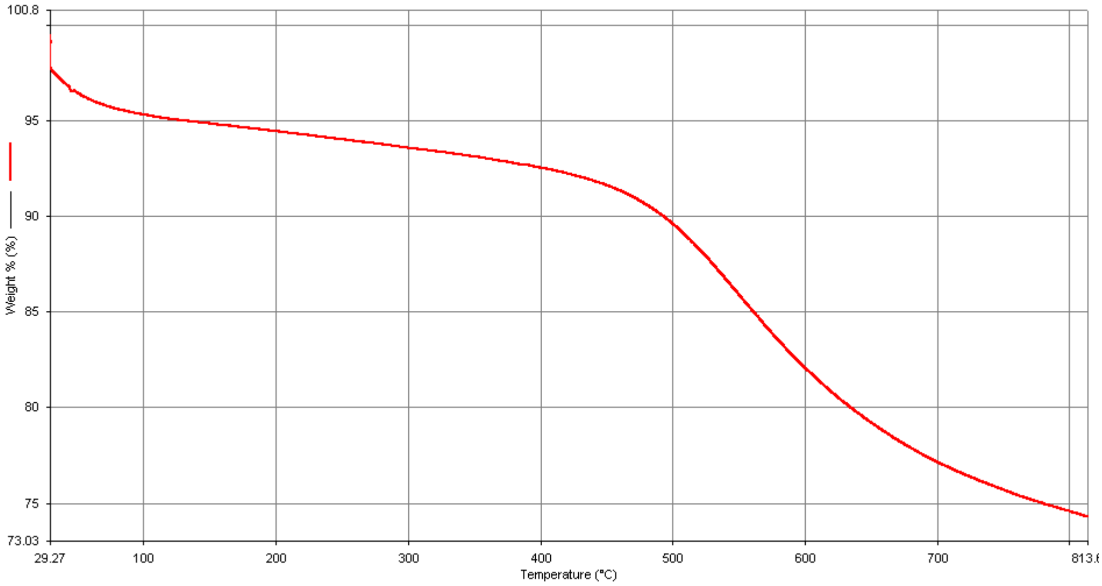


Figure 4.15: Thermal analysis of RHAC (temperature)

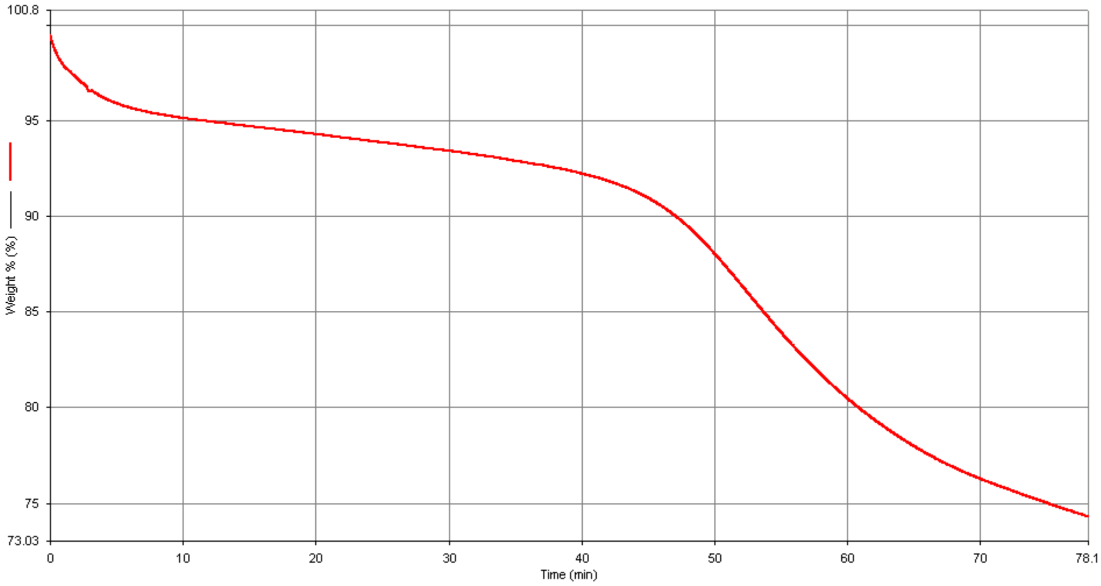


Figure 4.16: Thermal analysis of RHAC (time)

4.4.3 X-ray Diffraction (XRD)

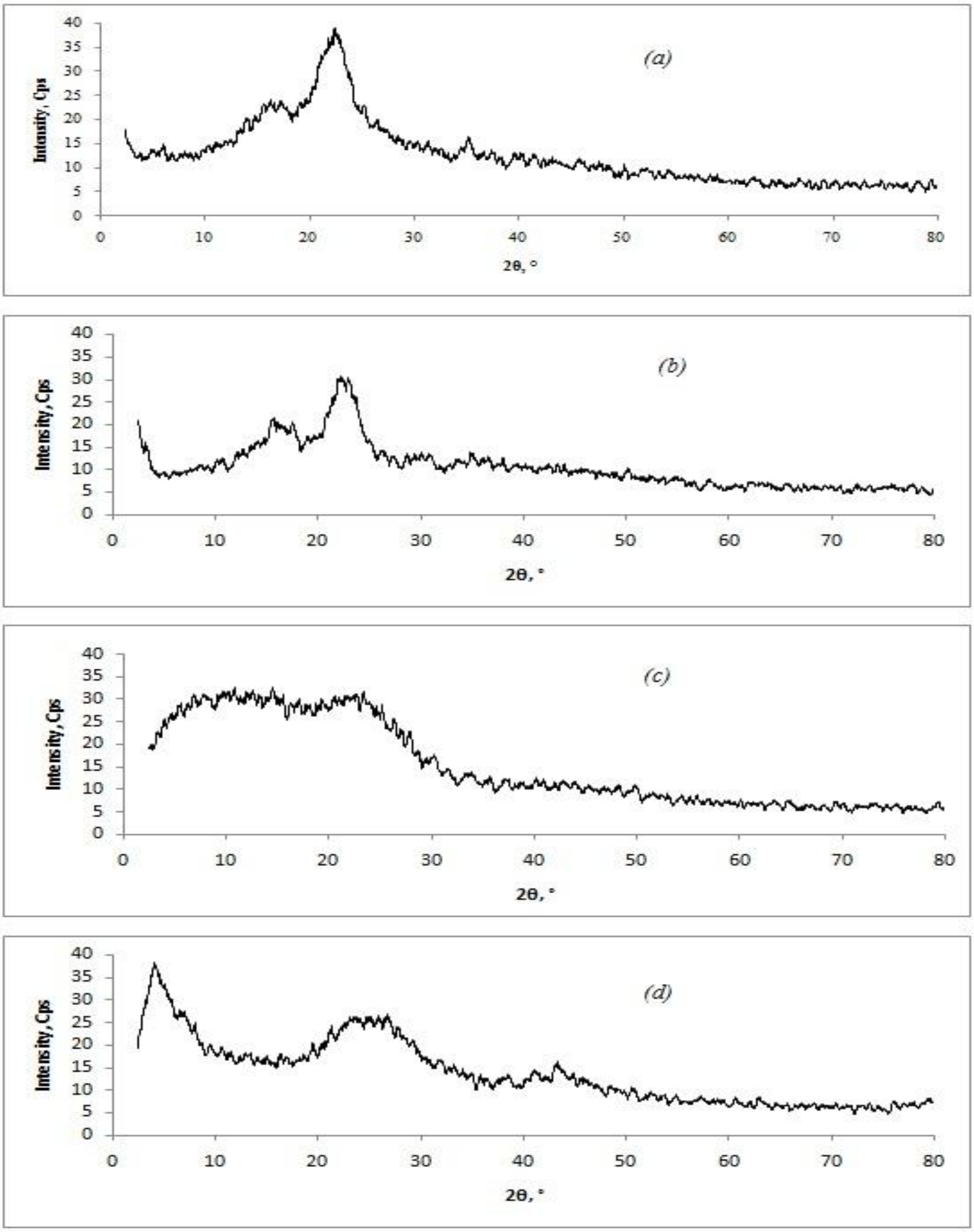


Figure 4.17: XRD curves of (a) RRH, (b) TRH, (c) RHAC and (d) commercial activated carbon, CAC

Figure 4.17(a), (b), (c), and (d) show the XRD curves for RRH, TRH, RHAC and commercial activated carbon (CAC) respectively. The XRD of the samples shown in Figure 4.17 contains a mixture of amorphous silica and carbon. XRD analysis commonly indicates crystallinity parts and amorphous component by sharp narrow diffraction peaks and broad (halo) peak respectively. The sharp peak observed from Figure 4.16(a) at 22° 2θ corresponds to the presence of completely crystalline silica. TRH sample from Figure 4.17(b) shows two diffused peaks which are broad peak at 16.5° 2θ and sharp peak at 2θ 22° which indicates a mixture of crystalline and amorphous phases of silica. The crystalline phases of silica are identified as crystobalite and tridymite (Yue et al, 2011). The XRD curves for RHAC and CAC samples show the presence of completely amorphous silica as broad peak is found from 15° to 22° 2θ and from 22° to 45° 2θ respectively. This analysis shows that CAC has similar high peak intensity with other samples which is approximately at 40 Cps.

4.4.4 Fourier transform infrared (FTIR)

Infrared spectroscopy provides information on chemical structure by identify the characteristic of functional groups. Figure 4.18 shows differences in the FTIR spectra of the samples. IR spectra of RRH and TRH samples show a wide band at about 3300-3700 cm^{-1}. The Si-OH, and O-H stretching mode of hexagonal groups and absorbed water can be assigned to this band. The position and asymmetry of this band at lower wavenumbers indicate the presence of strong hydrogen bonds. The peak around 1600 cm^{-1} of RRH, TRH and RHAC samples can be assigned to aromatic ring of C=C stretching vibration. The relative intensity of this peak was observed to be increased in RHAC which indicates the development of carbonization process. After carbonization process of TRH, FTIR spectrum of RHAC sample shown in Figure 4.18(d) was different from RRH and TRH. This finding could be due to many bands that were disappeared indicating elimination of volatile matter. This result proves that carbonization helps to produce higher carbonaceous material in RHAC which leads to high adsorption capability of RHAC.

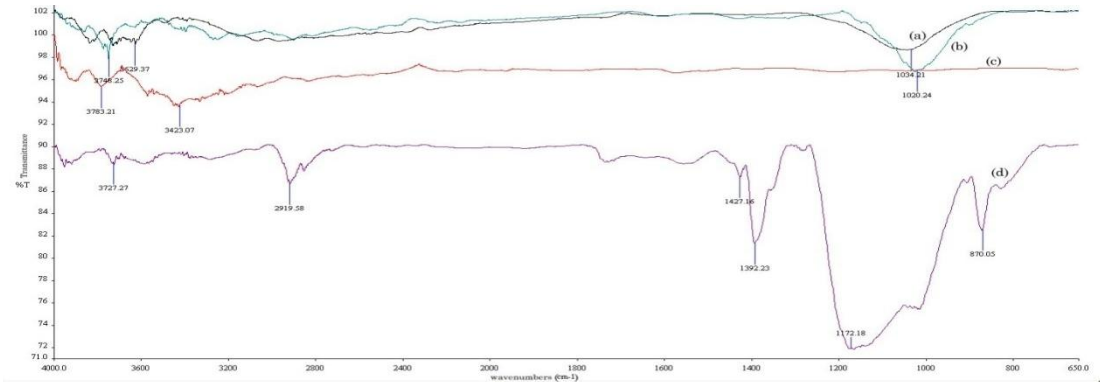


Figure 4.18: FTIR spectra of (a) RRH, (b) TRH, (c) commercial activated carbon, CAC and (d) RHAC

Table 4.3: RRH. TRH and RHAC spectrum and their possible assignment

Band position	Possible assignments
3629-3783	Free hydroxyl Si-OH, O-H stretching
2919-3423	C-H stretching
1392-1427	CH3 symmetric bending
1600	C=C stretching
1034-1172	C-O stretching

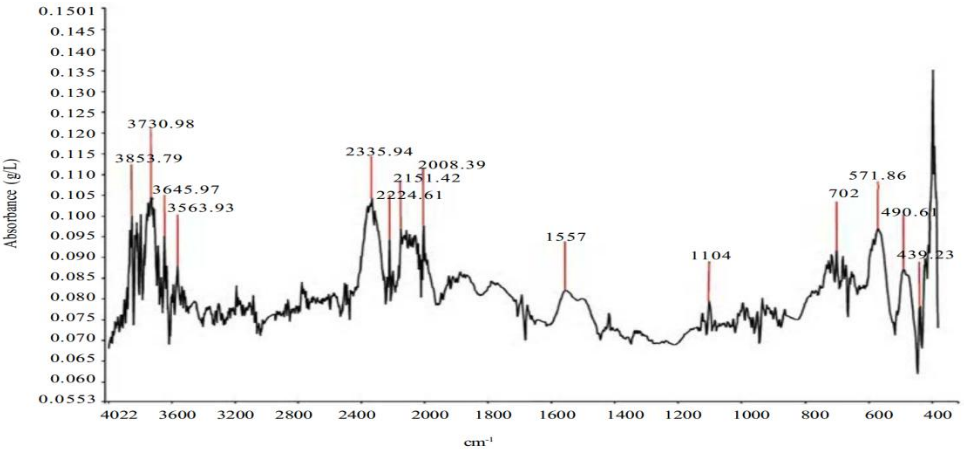
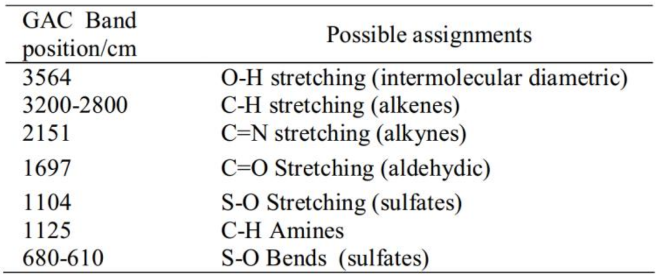


Figure 4.19: FTIR spectra of palm shell activated carbon (Onundi, 2010)



GAC Band position/cm	Possible assignments
3564	O-H stretching (intermolecular diametric)
3200-2800	C-H stretching (alkenes)
2151	C=N stretching (alkynes)
1697	C=O Stretching (aldehydic)
1104	S-O Stretching (sulfates)
1125	C-H Amines
680-610	S-O Bends (sulfates)

Figure 4.20: Peaks from GAC spectrum and their possible assignment (Onundi, 2010)

4.5 ADSORPTION STUDY

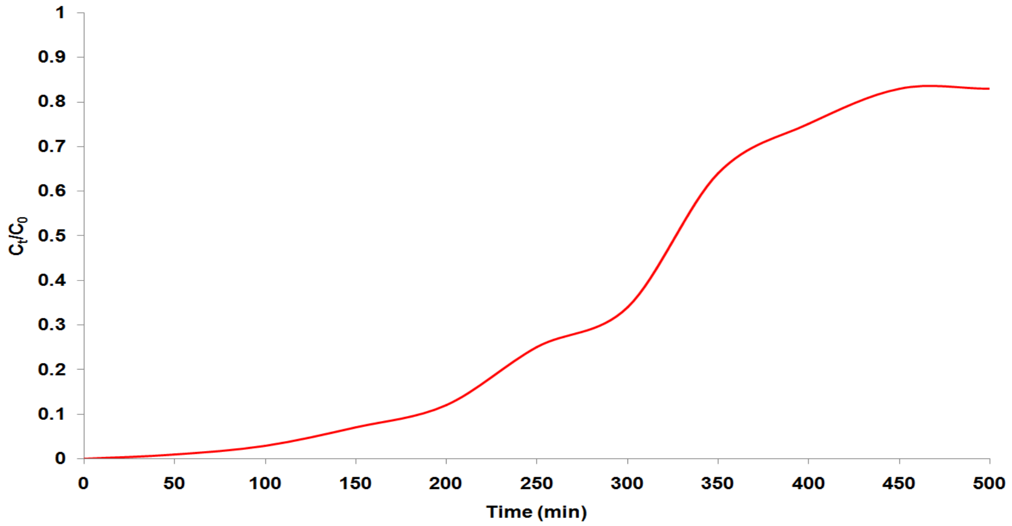


Figure 4.21: Breakthrough curves of Ni (II) sorption on RHAC

Table 4.4: Experimental data

Time (min)	C_t/C_0	Volume Effluent (ml)	Trapezoid
0	0	0	22.5
50	0.009	500	95
100	0.029	1000	247.5
150	0.07	1500	525
200	0.14	2000	900
250	0.22	2500	1450
300	0.36	3000	2500
350	0.64	3500	3550
400	0.78	4000	4025
450	0.83	4500	4150
500	0.83	5000	
550	0.83	5500	
600	0.83	6000	
650	0.83	6500	
700	0.83	7000	
750	0.83	7500	
800	0.83	8000	
850	0.83	8500	
900	0.83	9000	
950	0.83	9500	
1000	0.83	10000	
		Area under curve =	**17465**

$$q_{total}=\frac{QA}{1000}=\frac{(10)(17465)L}{1000\text{ ml}}\cdot\frac{mg}{L}\cdot\frac{ml}{min}=174.65\text{ mg}$$

Where;

q_{total} = maximum column capacity (mg)

C_{ad} ($C_{ad} = C_0 – C$) (mg/l)

t_{total} = total flow time (min)

Q = volumetric flow rate (ml/min)

A = area under the breakthrough curve

m = total dry weight of RHAC in column (g)

The equilibrium uptake (q_{eq}) is calculated as shown from Equation (2):

$$q_{eq}=\frac{q_{total}}{m}=\frac{174.65\text{ mg}}{4.25\text{ g}}=41.09\ \text{mg/g}$$

The total amount of Ni (II) sent to the column (W_{total}) is calculated as shown from Equation (4):

$$W_{total}=\frac{C_0Qt_{total}}{1000}=\frac{(100\ \text{mg/L})(10\,\text{ml/min})(450\text{ min})L}{1000\text{ ml}}=450\text{ mg}$$

Total removal percent of Ni (II) is the ratio of the maximum capacity of the column (q_{total}) to the total amount of Ni (II) sent to the column (W_{total}).

$$Y=\frac{q_{total}}{W_{total}}\times 100=\frac{174.65\text{ mg}}{450\text{ mg}}\times100=38.81\ \%$$

Table 4.5: Column data parameters obtained from different type of adsorbents a) RHAC at 500°C, b) WPOOF (waste pomace of olive oil factory) and c) tea factory.

Type of adsorbent	**Inlet Conc. (mg/L)**	**RHAC Bed Height (cm)**	**RHAC Weight (g)**	**Q (ml/min)**	**q_{total} (mg)**	**q_e (mg/g)**	**Y (%)**
a)RHAC	100	6.2	4.25	10	174.65	41.09	38.81
b)WPOOF	100	10	-	10	78.26	3.33	21.74
c)Tea factory	100	10	3.5	10	-	10.57	30.8

Figure 4.21 shows the breakthrough curve obtained for Ni (II) adsorption on the RHAC at bed heights of 6.2 cm, flowrate of 10 ml/min and Ni (II) inlet concentration of 100 mg/l. At the interval of 450 min, the value of C_t/C_o reached 0.83. Similar trends were obtained in literature for removal of Cr (IV) from WPOOF (waste pomace of olive oil factory) and removal of Ni (II) from tea factory (Malko and Nuhoglu, 2006). This value indicates the end of mass transfer zone or exhaustion point. When this has occurred, no more adsorption can take place in the bed.

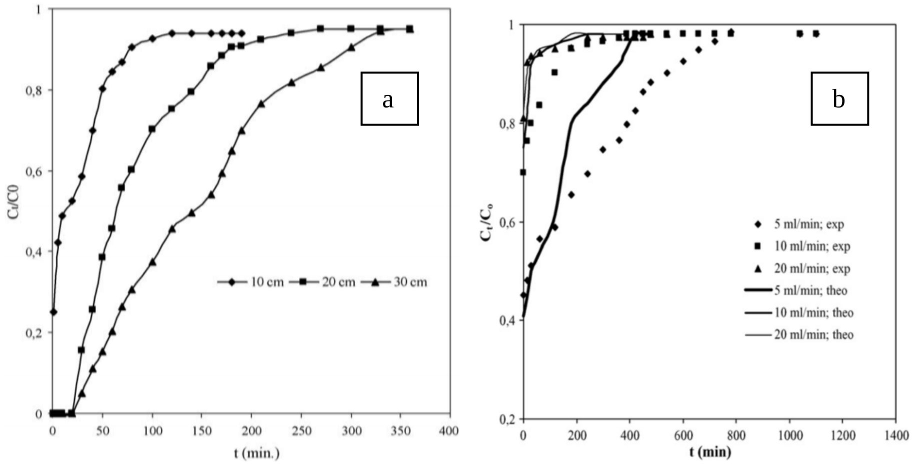


Figure 4.22: (a) Breakthrough curves of Cr (IV) sorption on WPOOF and (b) Ni (II) sorption on tea factory.

Table 4.5 shows that the adsorption capacity of RHAC is 41.09 mg/g which is higher compare to the other adsorbents. By comparing breakthrough curve of RHAC in Figure 4.20 with Figure 4.22 (a)(b), the breakthrough time increased with increasing the bed height. The bed height increased, the heavy metal had more time to contact with adsorbent that resulted in higher removal efficiency of heavy metal in the column (Yahaya, 2011). Higher bed column results in a decreased in the solute concentration in the effluent at the same time. The slope of breakthrough curve was slightly decreased with increasing bed height, which resulted in a broadened mass transfer zone. (Malko and Nuhoglu, 2006) states in their thesis that the Ni(II) uptake capacity of waste tea increased with the increase in bed height due to availability of more binding sites for sorption. Referring to table 4.4, it shows different result where RHAC has higher removal percent at low bed height. This result shows that RHAC has higher adsorption potential compare to other adsorbents at low bed height and shorter breakthrough time.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

As the production of rice husk increase, and the world now have major problem on producing low-cost commercial activated carbon, this project is expected can give new alternative to get new source of activated carbon. This new adsorption system that uses several factors that affecting adsorption, it is expected can give high adsorption capacity compare to the past project. Experimental isotherm studies will be used to describe this adsorption capacity.

This project has completed the first stage which is the production of rice husk-based activated carbon. This project study will continue to the second stage where adsorption activities will be observed by using fixed bed column. Based on the results obtained throughout the study, the conclusion has been made where the pores of rice husk can be made through grinding process, treatment process, and carbonization process. As there is reduction in weight observed during treatment and carbonization process, it shows that certain elements has been removed from rice husk and fixed the carbon content in RHAC (rice husk –based activated carbon). The bed capacity of 41.09 mg/g was obtained using 10 mg/l inlet Ni (II) concentration, 6.2 cm bed height and 10 ml/min flow rate. The Ni (II) removal percent was 38% which higher compare to other adsorbent. From these results, rice husk-based activated carbon has the potential as adsorbent for Ni (II) removal from aqueous solution.

There are several recommendation have been identified to further improve the current project study for future used. In order to justify the relation between adsorption potential of RHAC and the surface properties, surface area and pore sizes should be determine using BET method.. Adsorption activities can be investigated by study the effect of amount of activated carbon used, initial concentration of heavy metal ion, and investigate the affinity of AC-based rice husk towards different heavy metal ions using fixed bed column.

CHAPTER 6

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CHAPTER 7

APPENDICES

APPENDIX I

Procedure of making 1.0M of Sodium Hydroxide solution:-

Calculate molecular weight of sodium hydroxide:

NaOH = 23 + 16 +1 = 40 gram/mole

As 1.0M = 1.0 mole/liter, then dissolve 40 gram of NaOH in 1 liter flask of distilled water.

Procedure of making 100 ppm of Ni (II):-

Calculate grams of Ni (II) in $Ni(NO_3)_2$ for 100 ppm of Ni (II):

$$mass\ of\ Ni\ (II) = \frac{100}{1000000}\ (ppm) \times 1000\ (ml) = 0.1\ g$$

Determine grams of $Ni(NO_3)_2$ required in a 100 ml volumetric flask:.

$$mass\ of\ Ni(NO_3)_2\ = \frac{MW\ of\ Ni(NO_3)_2}{MW\ of\ Ni\ (II)}\ \times\ mass\ of\ Ni\ (II)$$

$$mass\ of\ Ni(NO_3)_2\ = \frac{182.71\ g/mol}{58.69\ g/mol} \times 0.1\ g = 0.31g$$

APPENDIX II


Figure 7.1: Raw Rice Husk


Figure 7.2: Oven used for drying process

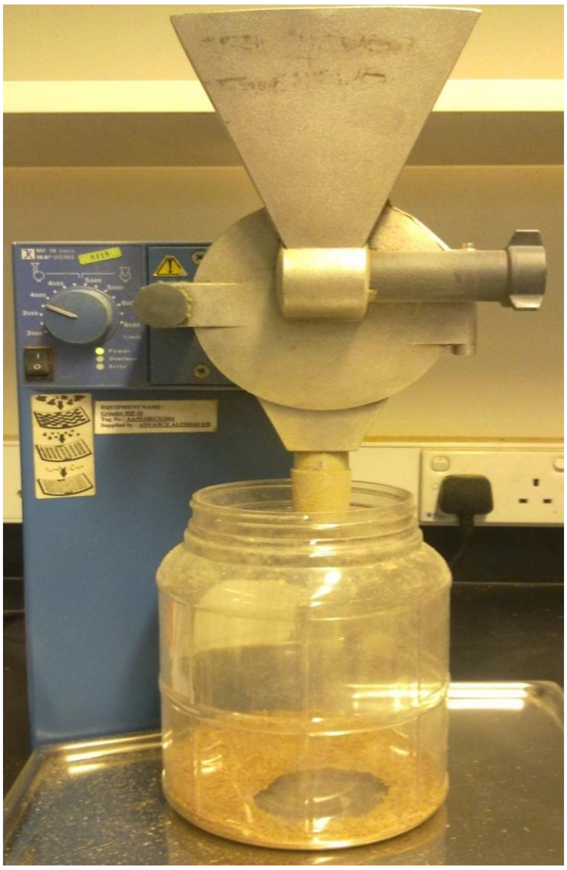


Figure 7.3: Grinder used for grinding process

Figure 7.4: Grinded Rice Husk

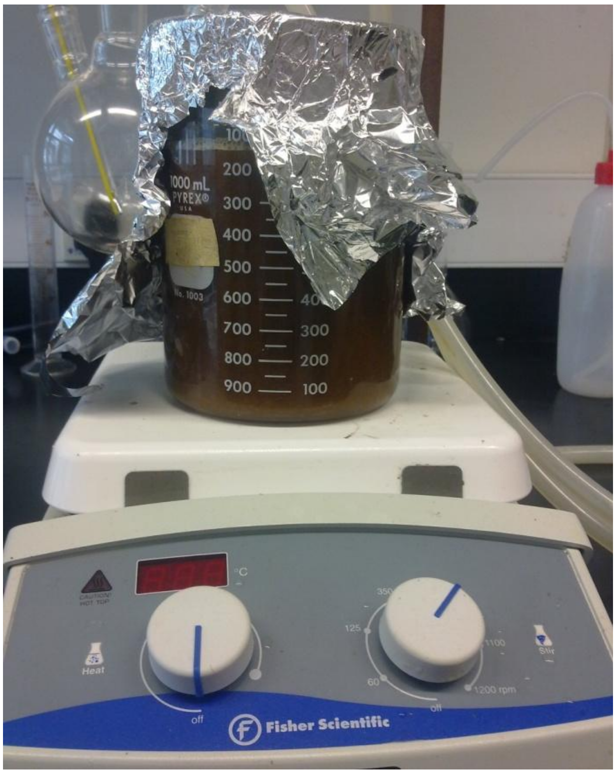


Figure 7.5: Raw Rice Husk treated with NaOH

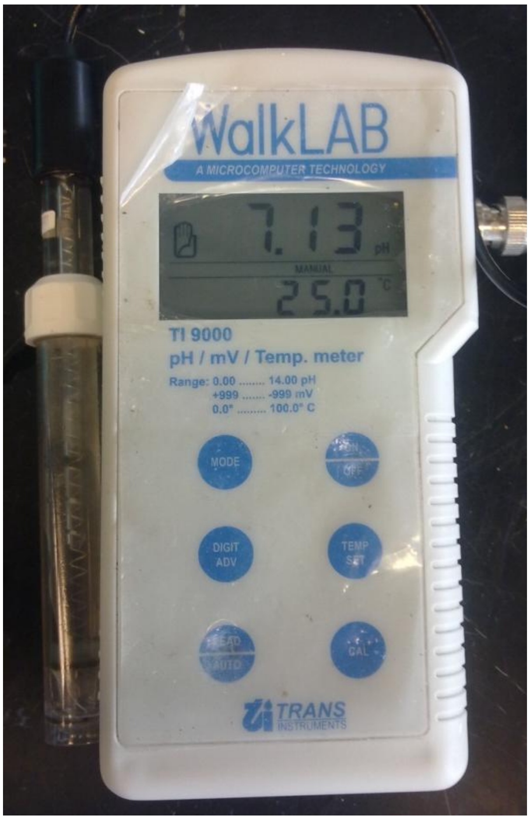


Figure 7.6: pH meter

Figure 7.7: Crucible containing TRH

Figure 7.8: Fixed Bed Activation Unit

Figure 7.9: Fixed Bed Column

APPENDIX III

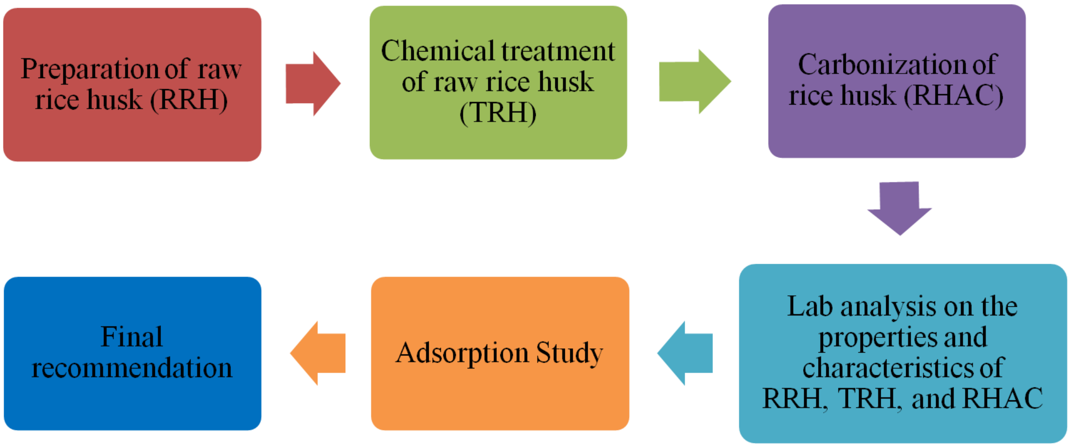
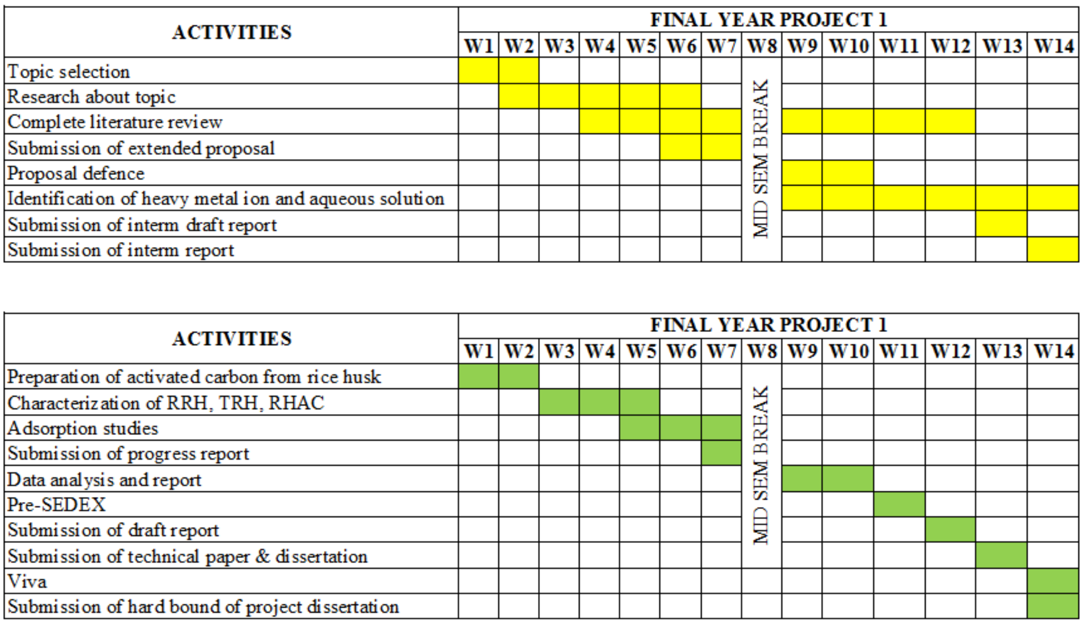


Figure 6.10: Flow chart of project activities



ACTIVITIES	FINAL YEAR PROJECT 1													
	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
Topic selection	🟨	🟨												
Research about topic		🟨	🟨	🟨	🟨	🟨		MID SEM BREAK						
Complete literature review				🟨	🟨	🟨	🟨		🟨	🟨	🟨	🟨		
Submission of extended proposal						🟨	🟨							
Proposal defence									🟨	🟨				
Identification of heavy metal ion and aqueous solution									🟨	🟨	🟨	🟨	🟨	🟨
Submission of interm draft report													🟨	
Submission of interm report														🟨

ACTIVITIES	FINAL YEAR PROJECT 1													
	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
Preparation of activated carbon from rice husk	🟩	🟩												
Characterization of RRH, TRH, RHAC			🟩	🟩	🟩			MID SEM BREAK						
Adsorption studies					🟩	🟩	🟩							
Submission of progress report							🟩							
Data analysis and report									🟩	🟩				
Pre-SEDEX											🟩			
Submission of draft report												🟩		
Submission of technical paper & dissertation													🟩	
Viva														🟩
Submission of hard bound of project dissertation														🟩

Figure 7.11: Gantt chart of project activities