CHAPTER 1: INTRODUCTION

1.1 Background Study

Rice husk is crop residue generated as a byproduct during dehusking at rice mills. There are about a fifth of the rice annual gross of 545 million tons in the world. There are about 400,000 tonne of rice husk produced by Malaysia annually. Common practice of disposing rice husk is by burning, thereby producing CO_2 and releasing toxics contaminants and solid particles to the environment.

Removal of heavy metals from industrial wasterwater is of primary importance because they do not only cause contamination of water but are also toxic to many life forms. Furthermore, they can accumulate in the food chain possibly causing severe danger to human health. Since most of heavy metals are non-degradable and toxic (Calace et al., 2002), so the realistic option is by reducing the concentration to acceptable levels before discharging them into the environment. Otherwise, these toxic metals would case a threat to the public health. Some of the most dangerous metals concerned are zinc, chromium, iron, nickel and mercury, cadmium and lead (Kadirvelu et al., 2001).

This study concerned with adsorbing zinc as a method to remove the metal ion from the wastewater. Most zinc enters the environment as a result of human activities such as mining, purifying zinc, lead and cadmium ores, steel production, coal burning, galvanizing and form wastewater of pharmaceuticals, paints, pigments, insecticides cosmetics and industries (A.K. Bhattacharya 2006). Technologies generally used for the removal of heavy metals such as ion exchange, chemical precipitation, ultra-filtration, reverse osmosis, electrochemical dispersion etc do not seem to be highly feasible due to their high cost (Axtell et al., 2003). As a result, recent development has focused of more cost effective alternatives.

Adsorption is one of the physic-chemical treatment process found to be effective in removing heavy metals from aqueous solution using low cost adsorption (Bailey et al., 1999). Adsorption of heavy metal ion is widely used as a unit operation in the treatment of industrial wastewater.

Most commonly used adsorbents are untreated plants wastes such as tea leaf powder (King et al., 2006), rubber leaf powder (Hanafiah, 2006), papaya wood (Saeed et al., 2005), newspaper pulp (Chakravarthy et al., 2007) etc. The type of adsorbent is determined by their adsorption capacity, regeneration characteristics and physical properties of subsequent product.

Rice husk is an abrasive thin layer of rice skin which covers the edible rice kernel and contains about 20% silica and has been reported as a good adsorbent for many metals and basic dyes (Suemitsu et al., 1986). It can be further developed to be made into activated carbon which is use as an adsorbent in water purification of industrial water. Rice husk can be converted from a low value agriculture byproduct into cheap activated carbon. This would add value to the agricultural commodities and furthermore help reduce the cost of disposing the rice husk. It is easy to obtain the rice husk from developing countries which do not posses technology to manipulate this agricultural waste.

1.2 Problem Statement

Across the world today renewable water resources available per person are roughly half what they were 50 years earlier. One of the major causes for this pollution is the presence of heavy metals in the environment. These metals, unlike organic materials, are very hazardous to human health. Zinc is one the metals and is a toxic and hazardous material that is currently contaminating the environment. It can accumulate in the food chain if left untreated and would cause harm to human health including disorder in human biological system, fever and diarrhea.

Due to the ineffective or expensive cost of current wastewater treatment, researchers are currently finding a low cost method that can remove the zinc from the water. The new method should focus on using byproduct that are cheap and abundance in developing countries. One of the cheap byproduct available here is rice husk which is currently disposed by burning thereby creating pollution to the environment.

1.3 Objective

4.1 To produce activated carbon from rice husk

4.2 To remove zinc (II) ion from aqueous solution using activated carbon prepared from rice husk

1.4 Relevancy of the Project

Adsorption process is effective to remove the heavy metals in waste water treatment. The water produced from a good waste water treatment is useful in terms that it can be safely used and can avoid harms. This existing method of waste water treatment has several disadvantage and furthermore the cost of implementation is very high, therefore using low cost adsorbents is found to be an effective alternative. Activated carbon can serve as a good low cost adsorbent and can be prepared using rice husk which is abundance here in this country. Current researches are focusing on finding methods to produce the most efficient activated carbon.

1.5 Feasibility of the Project within the given time frame

A number of studies on converting rice husk to activated carbon have been reported, as much as many information about the project can be obtained from the studies especially journals and reference books. All equipment and chemicals are already available in the Chemical Engineering Laboratory. With all the resource needed available, this project can be considered feasible within the time frame given.

CHAPTER 2: LITERATURE REVIEW

2.1 Water Pollution

Across the world today renewable water resources available per person are roughly half what they were in 1960 and this figure is expected to drop by half again by the year 2025 according to estimation by the World Bank. Clearly, if the water resources are not better managed, they could present a burden on economic growth as well as potential danger to human health and the environment (Mishra et al., 1996). The presence of heavy metals in the environment is of major concern because of their extreme toxicity and tendency for bioaccumulation in the food chain even in relatively low concentrations. Heavy metals like Fe, Cu, Zn, Ni and other trace elements are important for proper functioning of biological systems but their deficiency or excess could lead to a number of disorders.

Zinc is one of the most important metals often found in effluents discharged from industries involved in acid mine drainage, galvanizing plants, painting, agriculture, manufacturing and are not biodegradable which causes it to travels through the food chain via bioaccumulation. Excess zinc would causes fever, diarrhea, gastrointestinal tract irritation in humans and extensive mortality may result in fist and other aquatic species from the discharge of zinc containing wastewater (M. Akhtar et al, 2010). Therefore there is a growing significant interest regarding zinc removal from wasterwater. Its toxicity levels for humans are 100–500 mg/ day. World Health Organization (WHO) recommended the maximum acceptable concentration of zinc in drinking water as 5.0 mg/l (S. Tunali and T. Akar, 2010).

2.2 Adsorption technique

Conventional technologies for the removal of heavy metal such as chemical precipitation, adsorption, electrolysis, ion exchange and reverse osmosis are often neither effective nor economical (A. K. Bhattacharya, S.N. Mendel and S.K. Das, 2006). Among the disadvantages found in these methods are incomplete metal removal, high reagent and energy requirements and generations of toxic sludge or waste product that require disposal and further treatment. Most of these method are often ineffective or uneconomical when the heavy metal concentration is high in the range 1-100 mg/L and the permissible concentration is less than 1mg/L (Holan and Volesky, 1994).

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid, forming a molecular or atomic film. Adsorption, ion exchange, and chromatography are sorption processes in which certain adsorbates are selectively transferred from the fluid phase to the surface of insoluble, rigid particles suspended in a vessel or packed in a column (http://www.chemeurope.com).

Adsorption is to be distinguished from absorption, a process in which atoms or molecules move into the bulk of a porous material, such as the absorption of water by a sponge. Sorption is a more general term that includes both adsorption and absorption. Desorption refers to the reverse of adsorption, and is a process in which molecules adsorbed on a surface are transferred back into a bulk phase. The term adsorption is most often used in the context of solid surfaces in contact with liquids and gases. Molecules that have been adsorbed onto solid surfaces are referred to generically as adsorbates, and the surface to which they are adsorbed as the substrate or adsorbent. The exact nature of the bonding depends on the details of the species involved, but the adsorbed material is generally classified as exhibiting physisorption or chemisorptions (B. E. Bent, 2008).

Physisorption or physical adsorption is a type of adsorption in which the adsorbate adheres to the surface only through Van der Waals (weak intermolecular) interactions, which are also responsible for the non-ideal behaviour of real gases. Chemisorption is a type of adsorption whereby a molecule adheres to a surface through the formation of a chemical bond, as opposed to the Van der Waals forces which cause physisorption (www.bookrags.com).

2.3 Rice Husk as a Low Cost Adsorbent

Adsorption on activated carbon has long been recognized to be one of the most effective methods for the removal of pesticides and organic compounds from aqueous solutions.

Unfortunately, commercial activated carbon prepared from peat, wood, and coal is expensive, thus leading to the search for low-cost, easily available materials for production of activated carbons (S. Maiti, S. Purakayastha and B. Ghosh, 2008).

As a result, recent research has focused on the development of cost effective alternatives using various natural sources and industrial wastes. Industrial wastes are potential low-cost adsorbents for metal removal since some of them displayed high ion exchange capability. Several researches have made significant contributions in this area, utilizing a number of materials including fly ash, sugar beat pulp, clay, chitosan, coconut coirpitch carbon, biomass, zeolites, rice bran, soybeans and cotton seed hulls, sunflower stalks, calcined phosphate, low-grade phosphate, red mud, tea leaves, natural zeolite, almond shells, olive stones and peach, sediments of rivers, peanut hull, bentonite, saw dust, baggage fly ash, banana and orange peels and carrot residue. (A.K. Bhattacharya, S.N. Mandal, S.K. Das, 2006).

Rice husk was selected due to its local availability in abundance, chemical stability and insolubility in water (M. Akhtar et al, 2010). The abundance of rice husk is well documented. According to the Malaysia Ministry of Agriculture, there are 408,000 tonne of rice husk produce in Malaysia annually (T.G. Chuah et al, 2005).

Rice husk contains abundant floristic fiber, protein and some functional groups such as carboxyl, hydroxyl and amidogen (Nakbanpote et al., 2007), which make the adsorption processes possible and it has been successfully used to remove colored component (Vadivelan and Kumar, 2005; Han et al., 2008), metal ions (Srivastava et al., 2006, 2009; Naiya et al., 2009b) from water. Its adsorption capacity can be increased by modifying its texture by means of chemical and/or thermal treatments (Hsu and Pan, 2007).

Typical composition of the rice husks used is 16% silica, 14% lignin, 36% cellulose, 22% hemicelluloses, 3% extractive materials and 9% moisture (Rahman et al., 1997). Constituent in the rice husk (Grist, 1975) include 9.02% moisture, 3.27% crude protein, 1.18% fats, 33.71% carbohydrates, 35.68% crude fibre, 17.14% ash, and some metal oxide which consist mainly of silica, Calcium Oxide, MgO, Sodium Oxide and FeO. Previous studies reported rice husk has a low calorific value 3585 kcal/kg and high ash content (Feng et al, 2004).

Table 1 Comparison of adsorption capacities of the adsorbents for the removal of Zn (II) with those of other adsorbents (A.K. Bhattacharya, S.N. Mandal and S.K. Das, 2006).

No	Adsorbent	Adsorption Capacities (mg/g)
1	Phosphatic clay	25.10
2	Sunflower stalks	30.73
3	Penicillium chrysogenum	11.11
4	Streptovericillium cinnamoneum	9.15
5	Peanut husks	13.08
6	Activated Carbon	31.11
7	Sugar beat pulp	17.78
8	Red mud	14.51
9	Baggage fly ash	13.21
10	Coffee residues binding with clay as adsorbent	13.40
11	Calcined phosphate	20.60
12	Low-grade phosphate	10.32
13	Fly ash	11.11
14	Low rank Turkish coal	1.66
15	Tannic acid immobilized activated carbon	1.23
16	Carbon aerogel	1.18
17	Carrot residues	29.61
18	Clarified Sludge	15.53
19	Rice Husk Ash	14.30
20	Activated Alumina	13.69
21	Neem bark	13.29

2.4 Preparing Activated Carbon from Rice Husk

Activate carbon are made by carbonizing and then activating a material rich in carbon. Different activated carbon oriented from agricultural byproducts has also been published (Gupta and Torres, 1998). Activated carbons are highly effective adsorbents partly because of their large surface area. The characteristics of a good activated carbon are that it has:

i. high adsorptivity capability
ii. the ability to maintain its properties under usage condition

iii. have a long storage life

There exist several steps of preparing activated carbon from rice husk. Initially the rice husk is washed using distilled water to remove volatile compound, dust and bugs. Second, the rice husk was dried. Afterwards, the rice husk was break into pieces. Another available method is by crushing or grinding the rice husk. Next, the rice husk was sieved. Among the size used by is 50 um, 60 um, 75 um and 90 um.

After sieved, the rice husk would be treated with chemicals either acid or base. Chemical treatment would increase the surface area of the rice husk by producing more pores. According to I. A. Rahman, J. Ismail and H. Osman, (1997), this was done because it is observed that hemicellulose and lignin are readily digested by HNO_3 after 2 h of treatment. The reaction observed was vigorous and exothermic. After 10 h of digestion, the amounts of hemicellulose and lignin are 0.8 and 0.5%, respectively. Further loss of lignin was observed after 12 h digestion. Interestingly, however, cellulose behaves differently when compared to hemicellulose and lignin. It is observed that 6% cellulose remained even after 12 h digestion. This result indicates a high degree of resistance of the residual cellulose in the husk towards acid digestion. Overall, it is evident that hemicellulose and lignin are readily digested into simple species while cellulose is only partially digested.

The digestion produced a highly porous cellulose-silica material which has a relatively good adsorptivity in removing metal ions such as Zn (II), lead etc (I.A. Rahman et al, 2004). The greater pore size would lead to a larger capacity of adsorption. The differential behavior in acidic and basic treatment may be caused by the presense of high affinity ligand in the biomass such as cyano and amino group on the surface (Micheal Horshall Jr. et al, 2004).

Then, rice husk is carbonized in a free oxygen condition or inert atmosphere in order to remove volatile organic constituent leaving behind highly porous carbonaceous materials (Rahman. et al., 2000). The final product of the activated carbon would be different depending on the early treatment process. The pore structure would then be scan using Scanning Electron Microscope (SEM).

2.5 Factors Affecting Adsorption

From the literature review there are several factors that affect the adsorption capacity. Among the important criteria are pH of the chemical agent, carbonizing temperature and the particle size.

2.5.1 Particle Size

According to Karthikeyan et al., (2004) the adsorption capacity of activated carbon increase as the particle size decreases. The smaller particle size thereby gives more surface area which in turn increase the adsorbent adsorption capacity.

2.5.2 pH of the chemical agent

pH plays an important factor in determining the adsorption capacity of the activated carbon. Unfortunately, there is no observable trend in determining whether the activated carbon has better adsorption capacity in acid or in base.

According to Y. Guo et al. (2002), Activated carbon prepared from rice husk is active in lower pH range. The maximum adsorption of Cr (VI) species on the activated carbon was found in the pH range below 5 and the adsorbed amount was found to be negligible in the pH value over 8. This pattern in observable in other trends also. The adsorption was decreased with pH which perhaps due to the increase of the negative charge on the carbon surface, thus the electrostatic force of attraction between the adsorbent surfaces.

Meanwhile, according to A.K. Bhattacharya et al. (2006), the removal efficiency was found to be highly dependent on hydrogen ion concentration present in solution. Maximum Zn (II) removal was obtained in the pH range of 5–7. For rice husk ash with an initial concentration of 25 mg/L of Zn(II), maximum 96.8% removal was obtain at a pH value of 5. The effect of pH can be explained considering the surface charge on the adsorbent material. At low pH, due to high positive charge density due to protons on the surface sites, electrostatic repulsion will be high during uptake of metal ions resulting in lower removal efficiency. With increasing pH, electrostatic repulsion decreases due to reduction of positive charge density on the sorption sites thus resulting in an enhancement of metal adsorption. At higher pH values OH ions compete for Zn (II) with the active sites on the surface of the adsorbents. The uptake of Zn (II) as a function of hydrogen ion concentration was examined over a pH range of 3–11. Meanwhile, according to M. Ajmal et al., (2003), the percentage adsorption increased as the pH of the solution was increased and reached to a maximum value at pH 12.

According to Neeta Sherma and Jatinder Singh (2008), the rate of adsorption is maximum at the pH of 8. The sorption rare is lower in acidic range because at low pH, due to high positive charge density there is electrostatic repulsion resulting in lower rate of adsorption (Bhattacharya et al, 2006). With increasing pH, the electrostatic repulsion decrease due to reduction of positive charge density on the sorption site of adsorbent resulting in increased rate of adsorption.

2.5.3 Carbonizing temperature

The temperature dependence of adsorption studies of Zn (II) by rice husk was previously studied for a temperature range of 600-900 ‘C. The higher the carbonizing temperature, more pores are created. But there is a limit to this trend whereby, at 900’C, the adsorption capacity begins to drop whereby afterwards it was shown that at 900’C, the rice husk frame began to crumble.

CHAPTER 3: METHODOLOGY

The following are the methodology agreed upon. The overall methodology of this study can be divided into four major parts:

1. preparation of raw rice husks
2. treatment of raw rice husks
3. carbonization of treated rice husks
4. adsorption study
5. equipments

The first three parts determine the process of converting rice husks to activated carbon while the last part emphasize on the adsorption study of Zn(II) ions by the activated carbon prepared.

3.1 Preparation of Raw Rice Husks

- i. 1000g of raw rice husks was washed with distilled water several times to remove all the contaminants.
- ii. Rice husk is then dried in the oven at 80ºC for 24 hours.
- iii. The dried rice husks are then grinded using mortar grinder.
- iv. Grinded rice husk was sieved using a set of electrical sieve to obtain 63μm size of rice husks. Samples were then weighted.
- v. Several tests were then conducted to determine the characteristics of the raw rice husk:
  - a. The sample was scanned using Scanning Electron Microscope (SEM) to determine the morphology of the sample.
  - b. To obtain the sample functional groups, the sample was test using Fourier Transform Infrared Spectroscopy (FTIR)
  - c. The sample was also sent for CHN analyzer to determine the composition of Carbon, Nitrogen and Hydrogen in the sample

3.2 Treatment of Raw Rice Husks

- i. 1.0 M Sodium Hydroxide (NaOH) was prepared.

ii. 70 g of 63µm rice husk was digested with 700 ml of 1.0 M NaOH in 1000 ml beaker for 24 hours.
iii. Digested samples were then filtered and wash with excessive distilled water until filtrate pH was 7.
iv. Samples were placed in oven for drying at 80’C for 24 hours
v. Samples were weighted
vi. Several tests were then conducted to determine the characteristics of the treated rice husk:
    a. Sample was scanned using Scanning Electron Microscope (SEM) to determine the morphology of the sample.
    b. To obtain the sample functional groups, the sample was analyzed by using Fourier Transform Infrared Spectroscopy (FTIR)
    c. Sample was analyzed by using Thermo Gravimetric Analysis (TGA) to determine the ash content in the rice husk sample.
    d. Sample was analyzed by using Surface Area and Porosity Analyzer (SAP) to obtain the surface area images and its pore size
    e. Sample was analyzed by using CHN analyzer to determine the composition of carbon, nitrogen and hydrogen in the sample

3.3 Carbonization of Treated Rice Husks

i. A 20 g of treated rice husk was weighted and put in the crucible.
ii. The crucible containing rice husks was placed in the furnace when the temperature reached nearly 400ºC and the condition is in N_2 flow.
iii. After 1 hour, furnace temperature was reduced to 30ºC before the samples are taken out.
iv. After that, samples are weighted and percentage loss of weight is calculated.
v. Step 1 to 4 is repeated for a temperature of 600ºC.
vi. Several tests were then conducted to determine the characteristics of the activated carbons obtained. The tests included:
    a. Sample was scanned using Scanning Electron Microscope (SEM) to determine the morphology of the sample.
    b. To obtain the sample functional groups, the sample was analyzed by using Fourier Transform Infrared Spectroscopy (FTIR)

c. Sample was analyzed by using Thermo Gravimetric Analysis (TGA) to determine the ash content in the rice husk sample.

d. Sample was analyzed by using Surface Area and Porosity Analyzer (SAP) to obtain the surface area images and its pore size

e. Sample was analyzed by using CHN analyzer to determine the composition of carbon, nitrogen and hydrogen in the sample

3.4 Adsorption Study

i. 8 samples of 15 ml of 100 ppm Zn^{2+} were prepared and mixed with 0.1 g activated carbon prepared at 400ºC.

ii. The test tubes were left in a stem block at 850 rpm.

iii. The test tubes were then left for 15, 30, 45, 60, 90 and 120 minutes.

iv. All the test tubes were then filtered using filter paper and the residual is analyzed using AAS to check the Zn^{2+} concentration left in the residual.

v. Steps 1 to 5 are then repeated using the activated carbon prepared at 600 ºC.

vi. Graphs were plotted to observe the effect of parameters varied on the adsorption activities.

List of Equipment and Chemicals

1. Atomic Adsorption Spectrophometer (AAS)
2. 63 um siever
3. Mortar Grinder
4. Oven
5. Nitrogen Flow Furnace
6. Scanning Electron Microscopy (SEM)
7. pH meter
8. Stem Block
9. CHN analyzer
10. Crucible
11. Thermo gravimetric analysis (TGA)
12. Fourier Transform Infrared Spectroscopy (FTIR)
13. Surface Area and Porosity Analyzer (SAP)
14. Sodium Hydroxide (NaOH)
15. Zinc Chloride (ZnCl$_2$)
16. Distilled water

CHAPTER 4: RESULT AND DISCUSSION

4.1 Preparation of Activated Carbon

Initially, the rice husk was taken from a factory at Taiping, Perak. Inititally, the color of the rice husk is yellow brown. Afterwards, the sieved rice husk was treated with NaOH, the color of the rice husk changed into dark brown. After the rice husk was carbonized at 400 ºC and 600 ºC, the color of the sample was black, which is an indicator that the sample has been carbonized. The sample were labeled accordingly

Table 2 Labels and description of samples

No	Label	Description
1	RRH	Raw rice husk
2	TRH	Rice husk treated with sodium hydroxide
3	CRH400	Rice husk carbonized at 400ºC
4	CRH600	Rice husk carbonized at 600ºC

Figure 1 Raw Rice Husk before grinding

Figure 2 Rice Husk after sieving

Figure 3 Treated Rice Husk

Figure 4 Activated Carbon

4.2 Surface Morphological Analysis

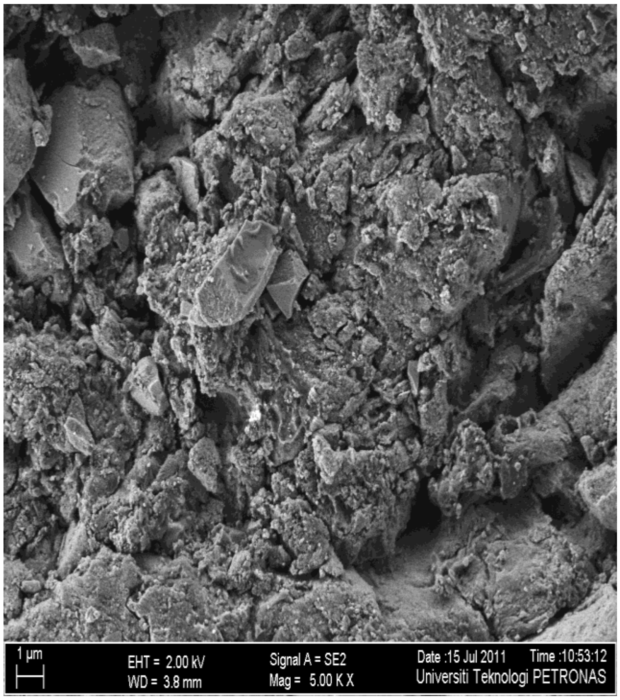

Figure 5 Raw rice husk

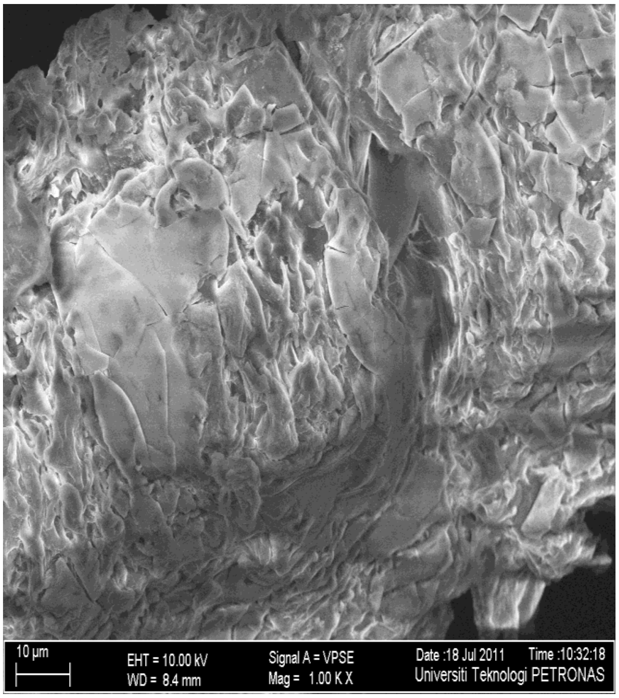

Figure 6 Treated rice husk

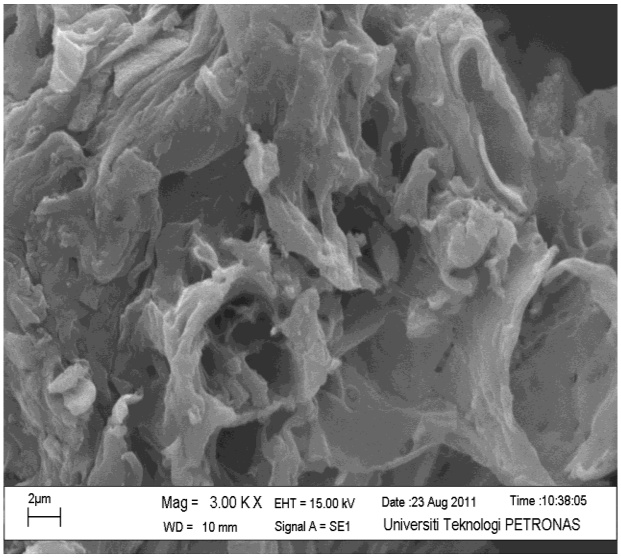

Figure 7 Rice husk carbonized at 400ºC

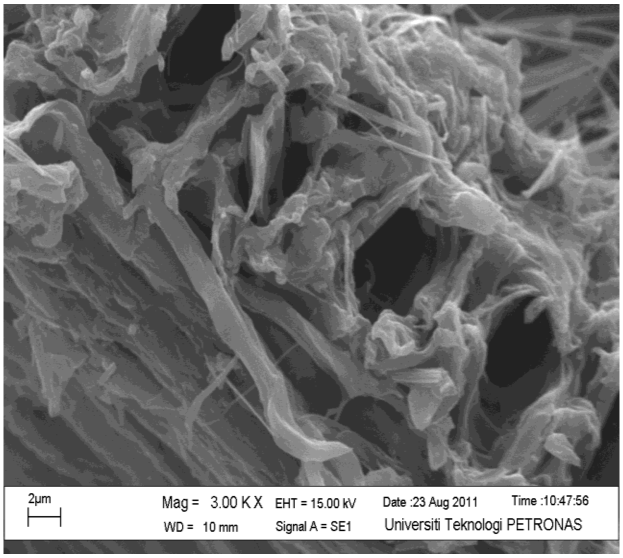

Figure 8 Rice husk carbonized at 600ºC

Figure 5 shows that initially there are no pores in the raw rice husk, afterwards pores formation is observed in the treated rice husk. Then, pores are observed and structured when turns into activated carbon.

This is explained by understanding that rice husk which contains high ash content which is mainly silica, can react with the treatment agent. T.H. Liou and S. J. Wu (2009) relate that the disappearance of silica simultaneously creates more surface area and new pore structure. Yenageh et al (2006) reported that the surface area of activated carbon depends on the ash content. Therefore, the lesser the ash content, the more surface area exist.

Figure 7 shows that when the treated rice husk is carbonized at 400ºC, more pores are created thereby increasing the surface area of the rice carbon. This is due to the volatile component are removed during the carbonization process. Hydrogen and Nitrogen has been known to be gaseous component and are easily removed, silica however are removed due to the silicon atom being attached to the oxygen atoms thereby making the component more volatile.

From figure 8, it is apparent there are the porosity is higher compared to in figure 7, whereby this shows that the carbonization temperature at 600ºC is better than at 400ºC. The higher the temperature, more volatile component is removed thereby creating more pores for the activated carbon. For the carbonizing temperature, there are maximum temperatures whereby the pores will collapse. T.H. Liou and S. J. Wu (2009) reported that the temperature is around 900ºC. The collapse happen due to the entire volatile component is already removed, thereby leaving the carbon and silica, in the form of pores, which in turn combine or collapse due to the heat absorbed.

4.3 CHN Analysis

Table 3 CHN Elemental Analysis result

CHNS Results		C	H	N
RRH	1	37.21	4.441	0.928
	2	36.73	4.480	0.912
	3	36.91	4.548	0.916
	Avg	**36.95**	**4.490**	**0.919**
TRH	1	44.46	7.502	0.671
	2	43.13	6.106	0.679
	Avg	**43.80**	**6.804**	**0.675**
CRH400	1	62.95	1.997	0.761
	2	51.87	2.829	0.794
	Avg	**57.41**	**2.413**	**0.778**
CRH600	1	65.78	3.020	1.055
	2	68.78	3.883	1.142
	Avg	**67.28**	**3.4515**	**1.099**

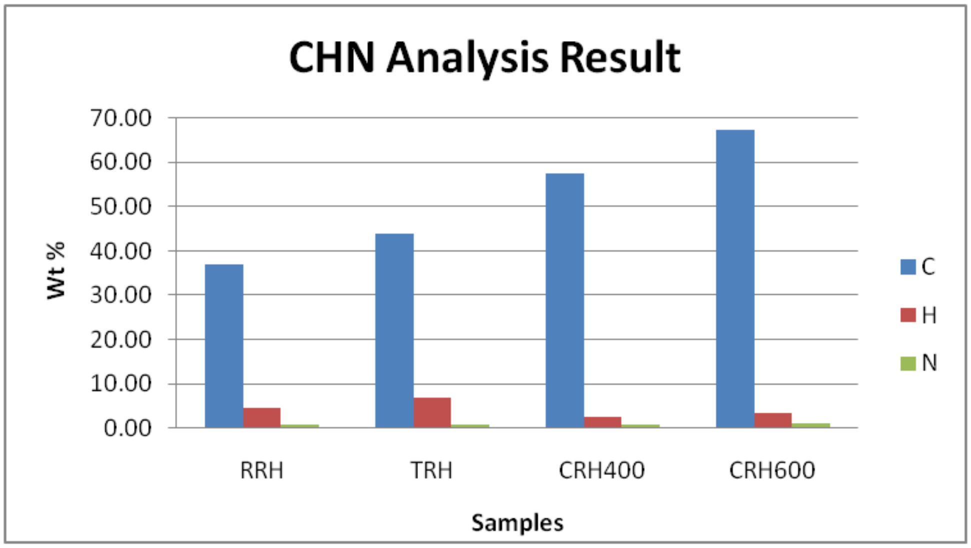

Figure 9 Graphical result of CHN Analysis

Grist, 1975 and Rahman et al., 1997, S.B. Dafalla, H. Mukhtar, M.S. Shaharun 2010, states that around 50% of the rice husk constituent is cellulose and around 33% of the rice husk constitute of carbohydrates. The CHN analysis results confirmed the findings. Figure 9 reveals that the main component of the rice husk is carbon followed by oxygen.

T. H. Liou and S. J. Wu (2009) reported that base treatment remove silica by reacting to form sodium silicate (Na_2SiO_3). The Na_2SiO_3 is soluble in water and is removed by adequate water-washing. The overall formation for the reaction is given as below:

$$2NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O \qquad (1)$$

This indicates that silica reacts with the activation agent. T. H. Liou, S. J. Wu (2009) also indicates that other elements in the rice husk are removed by simple water washing procedure. These observations shows that the chemical treatment, by diminishing the native metallic impurity, help prevent the pore obstruction that leads to a reduction in adsorption capacity.

The analysis reveals changes in the rice husk elements and ash content (SiO_2) before and after treatment and after activation. The analysis indicates that the percentage of carbon in the activated carbon is higher than that in un-carbonized rice husk. T. H. Liou, S. J. Wu (2009) states that the decrease in hydrogen and oxygen contents may be attributed to the formation of water vapor and carbon dioxide.

4.4 TGA Analysis

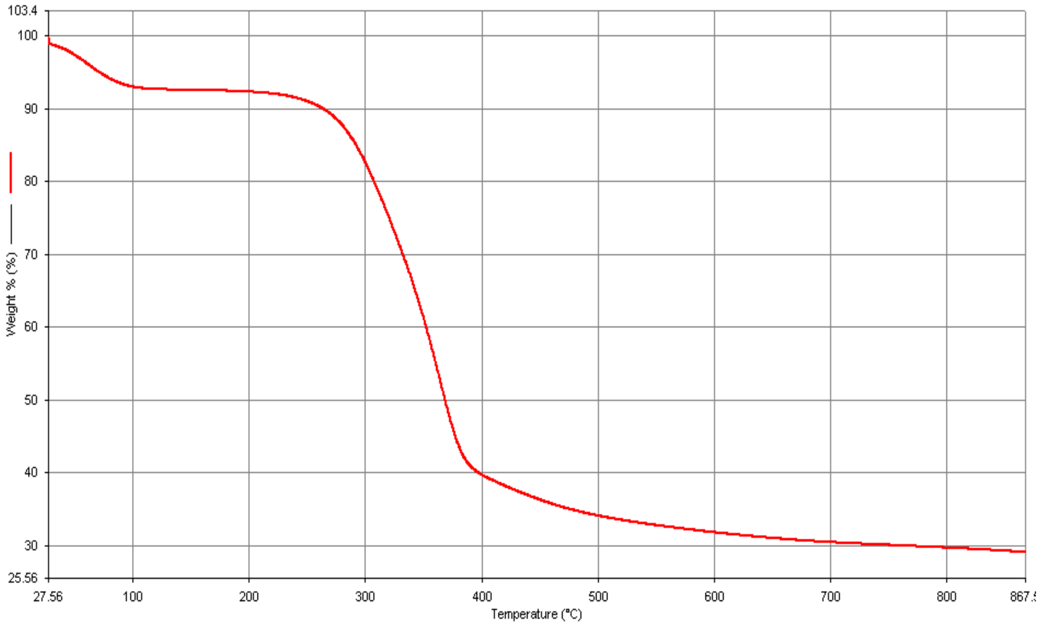

Figure 10 Raw rice husk

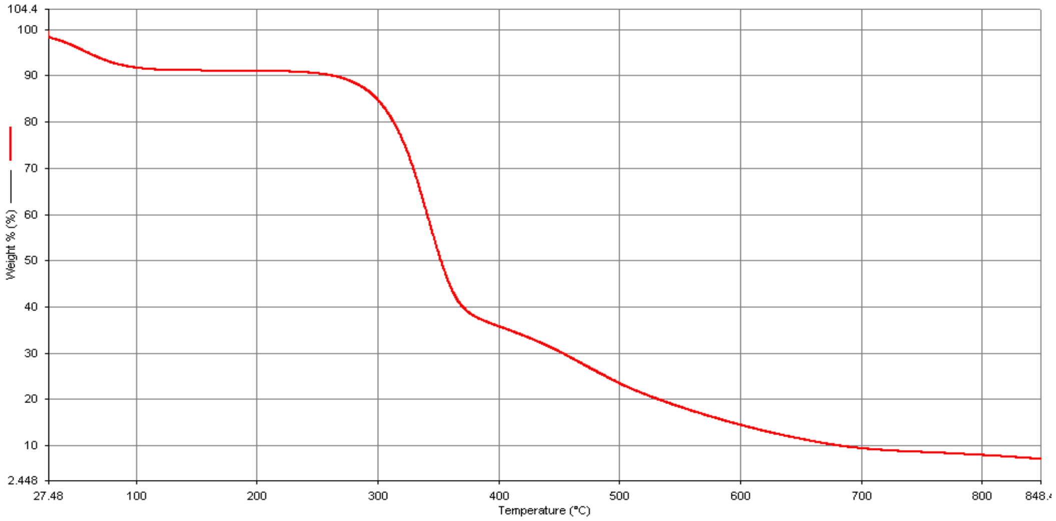

Figure 11 Treated rice husk

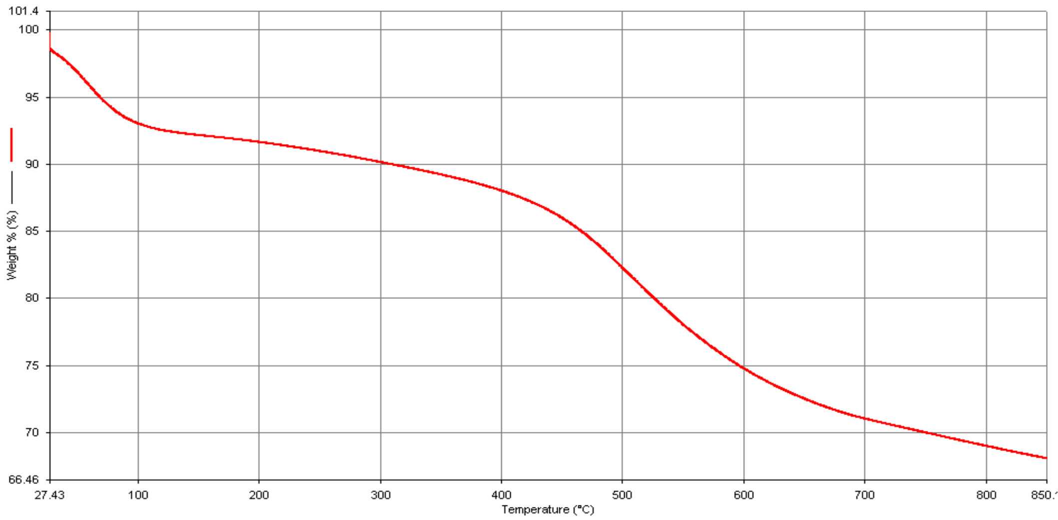

Figure 12 Activated Carbon at 400 ºC

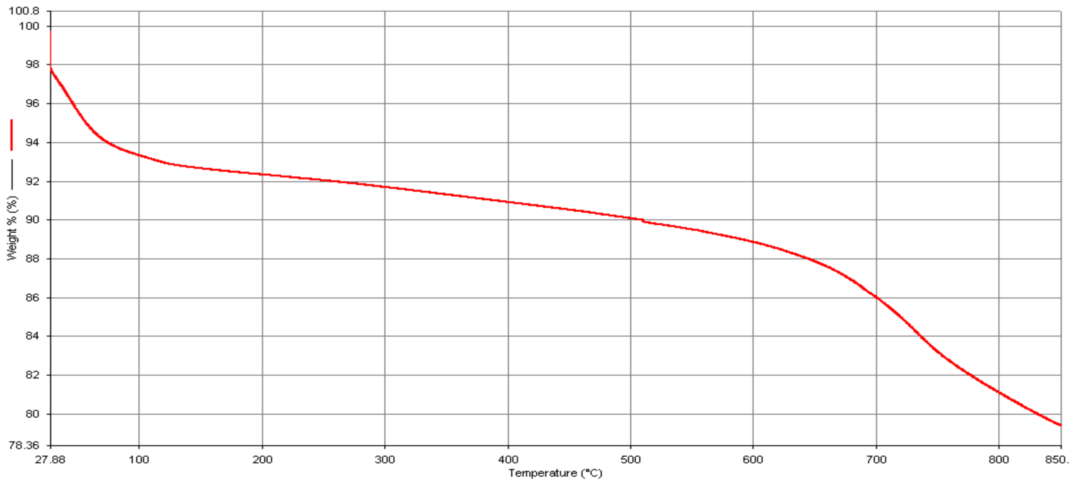

Figure 13 Activated Carbon at 600 ºC

From figure 11, shows the TGA curve of treated rice husk. The mass loss lower than 250ºC are attributed to the removal of moisture. The mass loss between 250 and 350ºC however are attributed to the decomposition of volatile materials. The mass losses above 350ºC meanwhile are attributed to the decomposition of the structure. This TGA analysis did not show a straight horizontal line which indicates that all the ash component has been removed. This is due to the time and temperature of the test need to be longer and higher for the straight line to appear.

Therefore we can conclude that, TGA analysis shows that the activation process can be divided into three stages. For the first stage, moisture is released thereby leaving only dried component of rice husk. During the second stage, volatile elements decompose and are remove in forms of volatile gaseous. The final stage is where the rice husk fully turns into activated carbon.

4.5 XRD Analysis

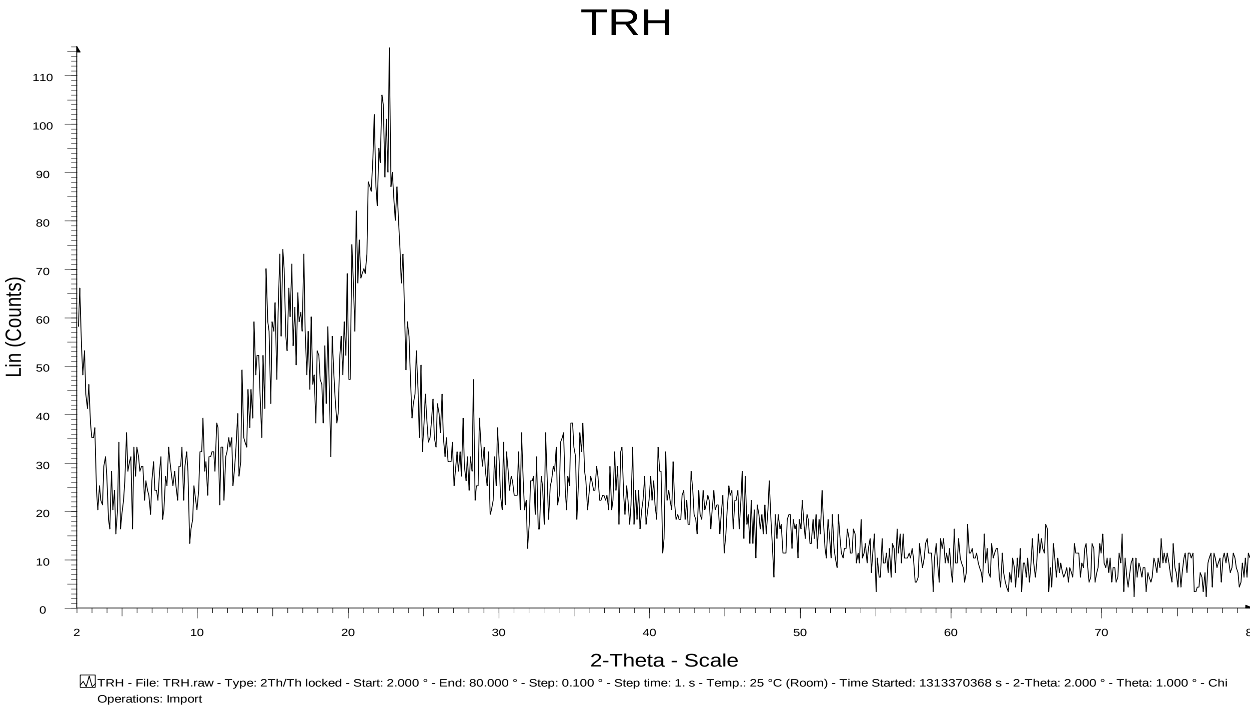

Figure 13 Treated Rice Husk

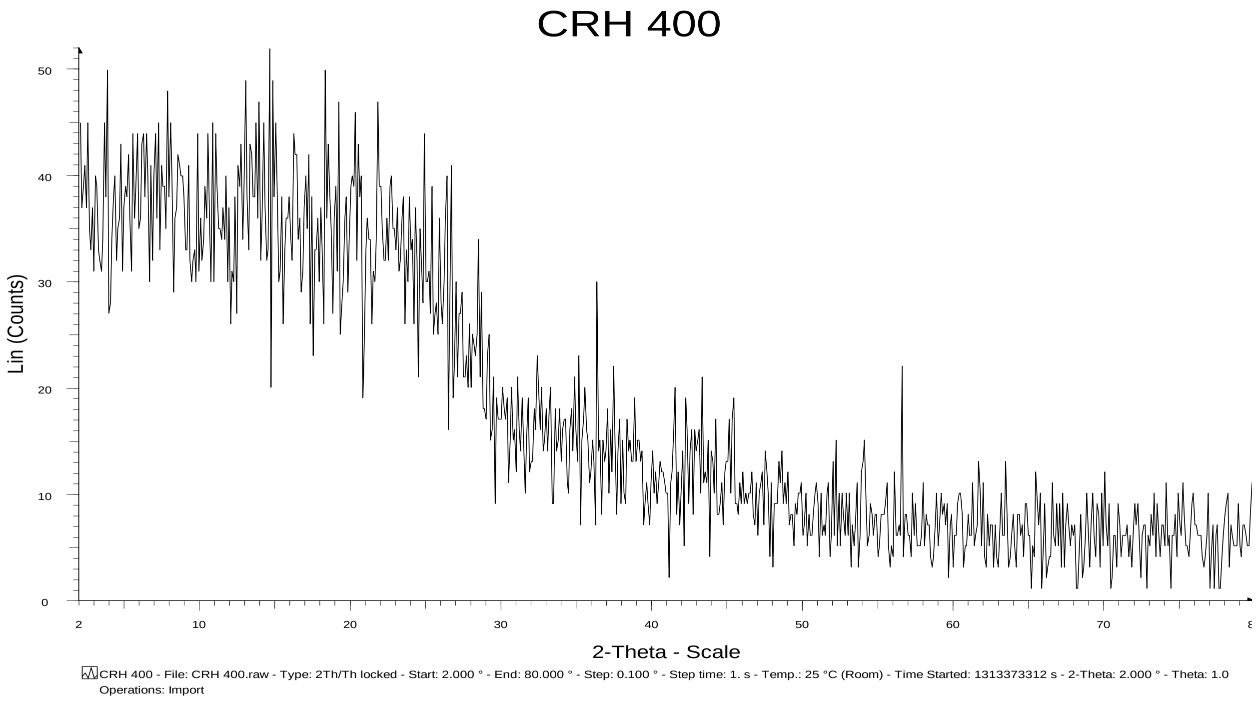

Figure 14 Rice husk carbonized at 400 ºC

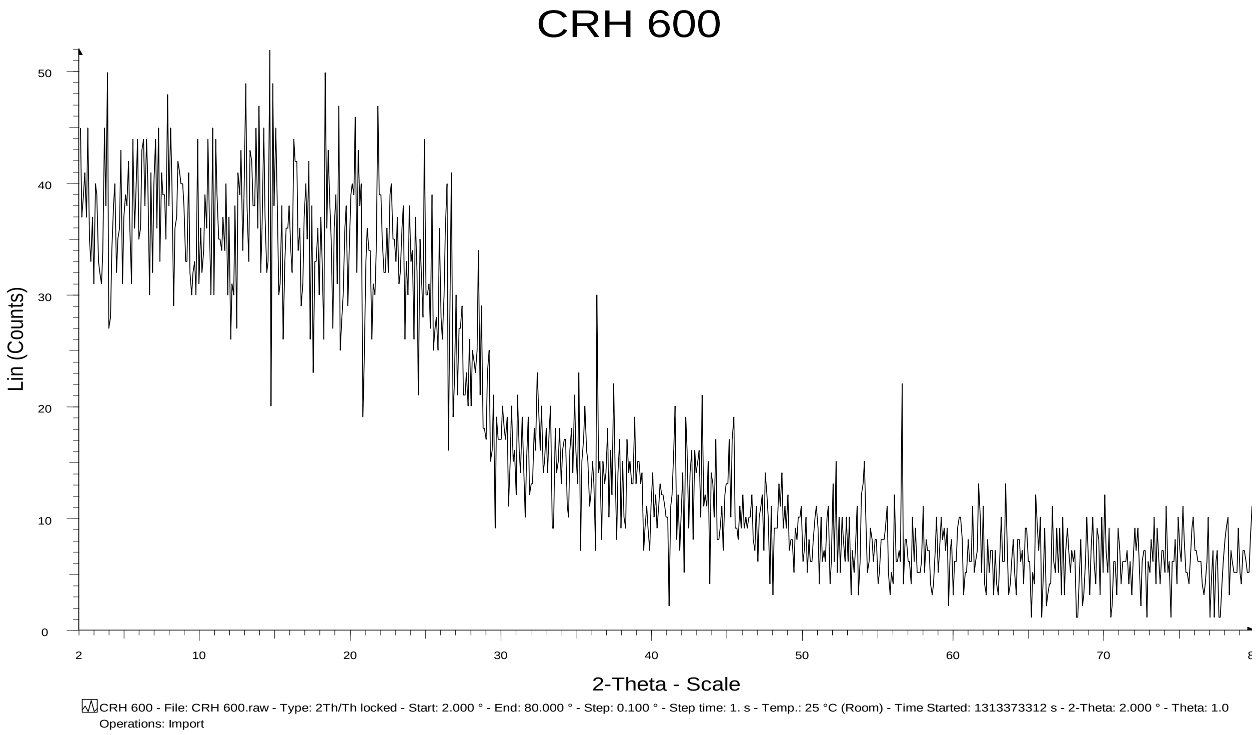

Figure 15 Rice husk carbonized at 600 ºC

Figure 13 to 15 shows the diffraction analysis of treated rice husk and activated carbon samples. From the picture it can be said that the diffraction peak around 2Theta=15-22.5º. This shows that the activated carbon produced in this project have similarities with the commercial activated carbon in the market.

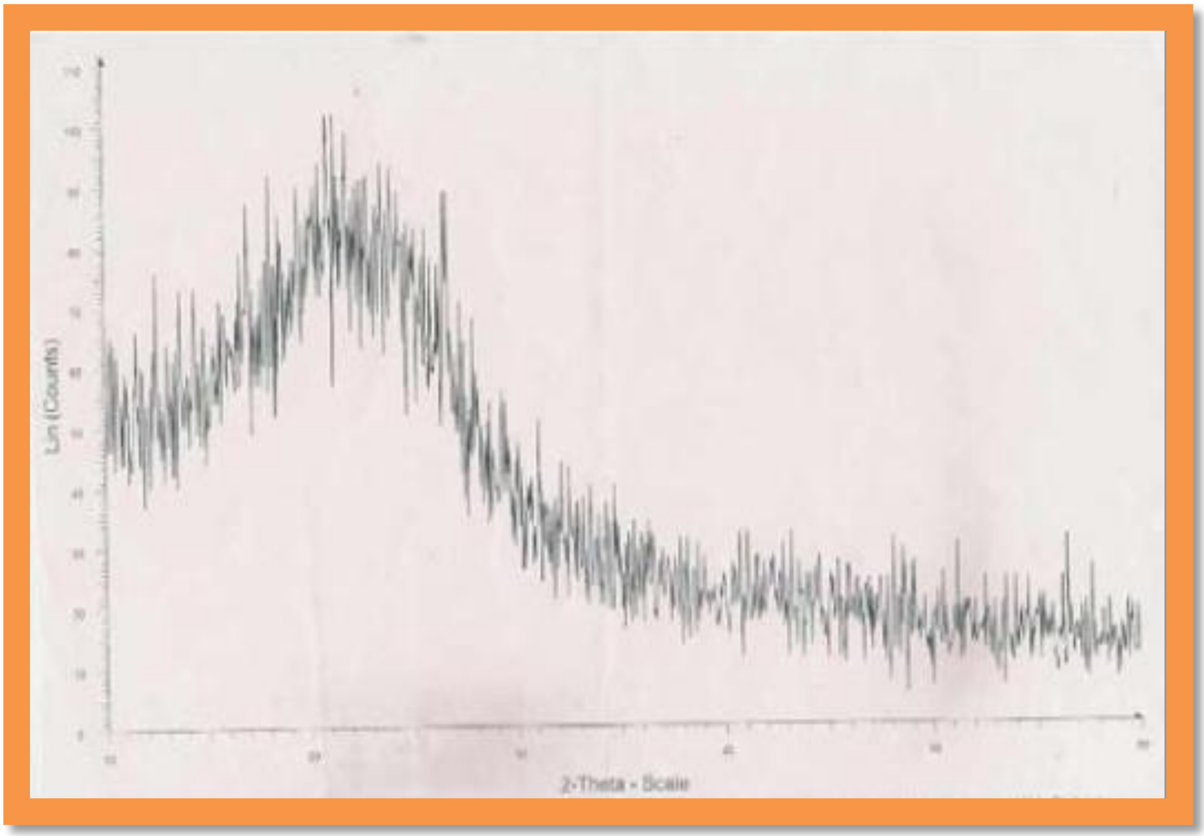

Figure 16 XRD pattern of the sorbents such as rice husk char, activated rice husk char and commercial activated charcoal gave the XRD pattern shown. (Tony et al, 2004)

4.6 FTIR Analysis

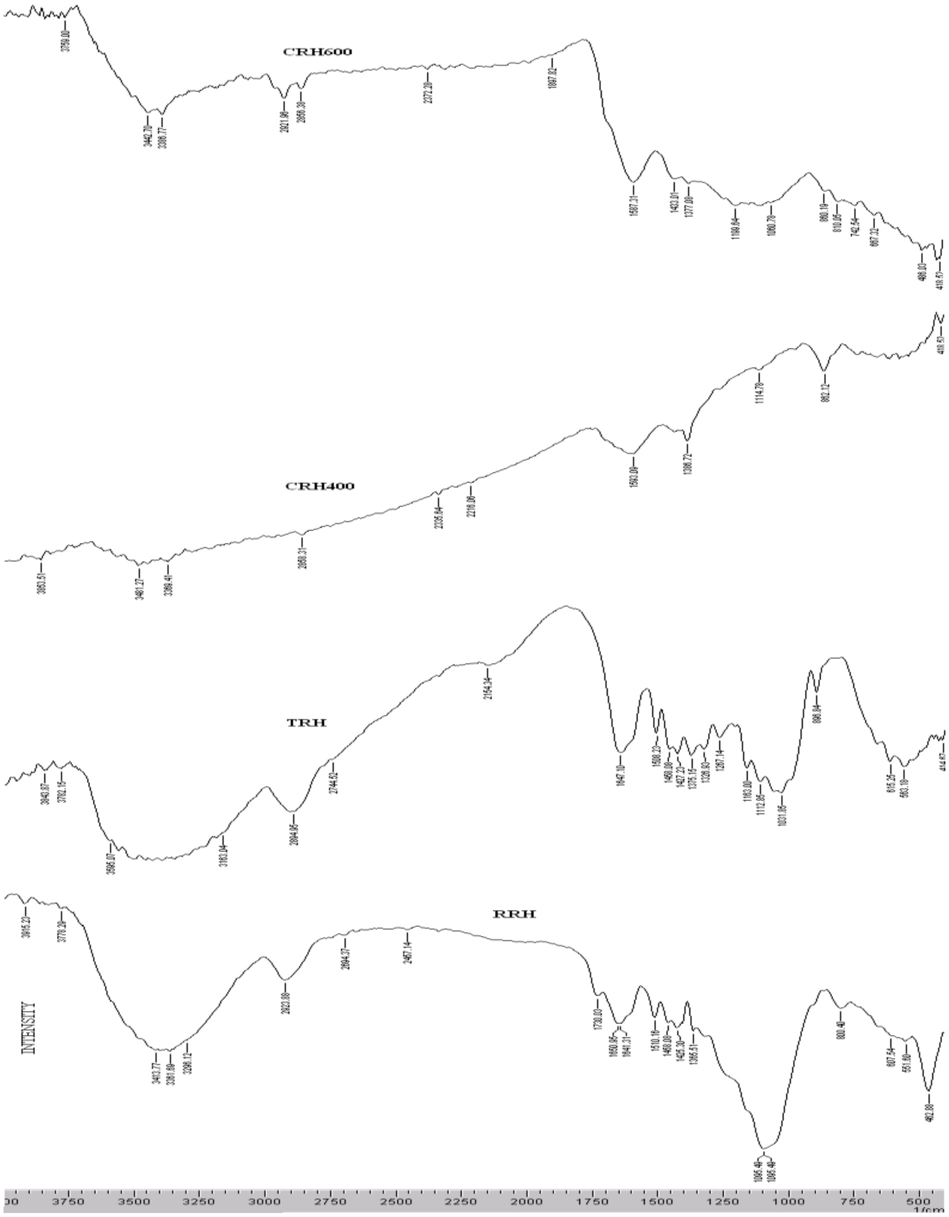

Figure 17 FTIR Analysis results

The band is located about 3400cm^{-1} is attributed to O-H hydroxyl groups. The band at approximately 2900cm^{-1} corresponds to the C-H vibrations. The band at 1700cm^{-1} is attributed to the ketones, aldehydes and carbonyl C=O groups. The band at about 1600 cm^{-1} may be assigned to aromatic stretching group C=C. A peak near 1400 cm^{-1} could be attributed to $–CH_2$ and $–CH_3$ vibrations. The band at 1200-1300cm^{-1} may be assigned to C-O vibrations in phenols, ethers and esters. The peak at $1500cm^{-1}$ can be attributed to R-OH, alcohol groups. The band from 400 to $1100cm^{-1}$ are probably due to the silicon atom attached initially to the oxygen in the rice husk. After treating the rice husk with chemical agent, some of the functional group disappeared. This is due to the functional groups reaction with NaOH and its subsequent removal by water. Next, we can find that after carbonizing the rice husk in a N_2 atmosphere, the spectrum of CRH sample is different from the raw material. Many bands disappeared after carbonization which indicates the vaporization of organic matter. This could be explained by the fact that in the carbonization process with N_2, moisture and volatile matters were removed from the material.

4.7 AAS Analysis

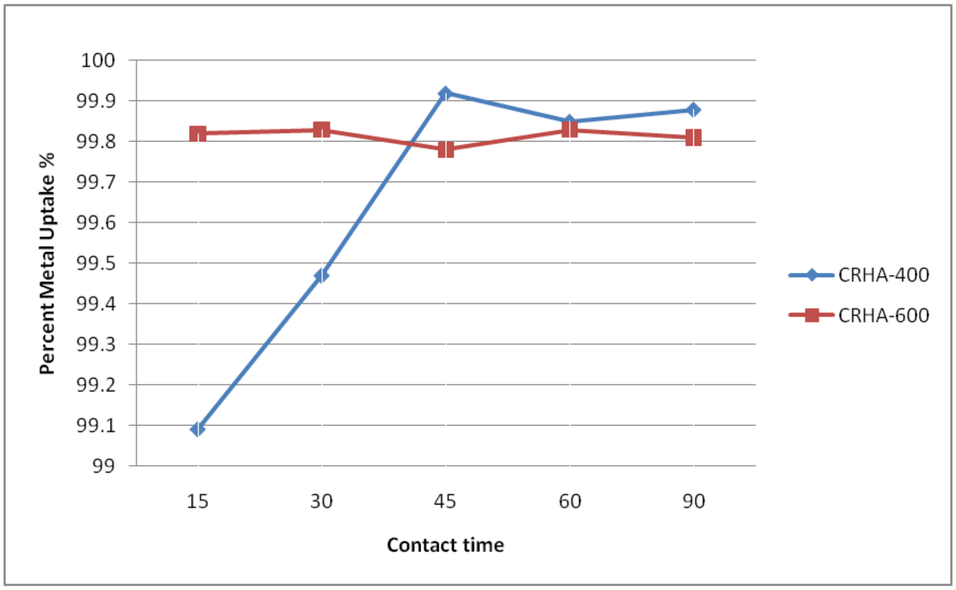

Figure 18 Percent Zn (II) ion uptake vs contact time

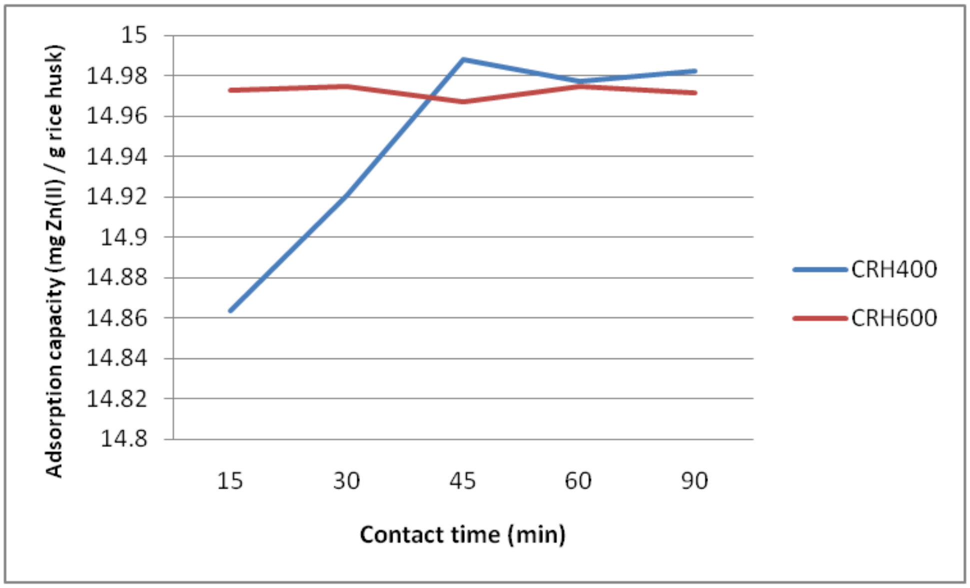

Figure 19 Adsorption capacity of activated carbon

Figure 18 shows that both activated carbon percent metal uptake is nearly 100% after 15 minutes. This result confirms the efficiency of activated carbon prepared from rice husk. N.S. Awwad et al, rice husk has been proven to remove 95% of La (III) metal ions on optimum conditions. Furthermore, we can say that increasing the contact time would increase the percent metal uptake until the adsorption reaches equilibrium.

From figure 19, it can be seen that CRH600 is reaches equilibrium faster than CRH400. This could be due to CRH600 has more pores compared to CRH400 thereby proving that CRH600 has better adsorption capability compared to CRH400. Therefore, the higher the carbonization temperature, the more pores the activated carbon possess thereby the higher its adsorption capability.

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

From the experiment, highly porous activated carbon was produced through grinding, chemical treatment and carbonization process. The activated carbon prepared from rice husk treated with sodium hydroxide was proven to be efficient in removing Zn (II) ions from aqueous solutions. The adsorption efficiency of the activated carbon produced is nearly 100%. Besides that, it is also found that the higher the carbonization temperature, the higher the adsorption capability of the activated carbon.

5.2 Recommendations:

1. Further experiment should study the effect of initial concentration of metal ion or dosage of rice husk activated carbon on adsorption study.
2. Time for TGA analysis should be prolonged to determine the weight of the ash content
3. Further experiment should focus on higher temperature to determine optimum and maximum temperature.
4. Further should use the Brunauer-Emmet-Teller (BET) method. Using BET results, the relation between adsorption capacity and the surface area properties can be justified.
5. To use shorter duration of time interval such as 2, 4, 6 and 10 minutes
6. To determine the adsorption capacity of other metal ion such as Lead and Nickel

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APPENDIX I

Figure 20 CHN Analyzer

Figure 21 AAS

Figure 22 TGA

Figure 23 FTIR

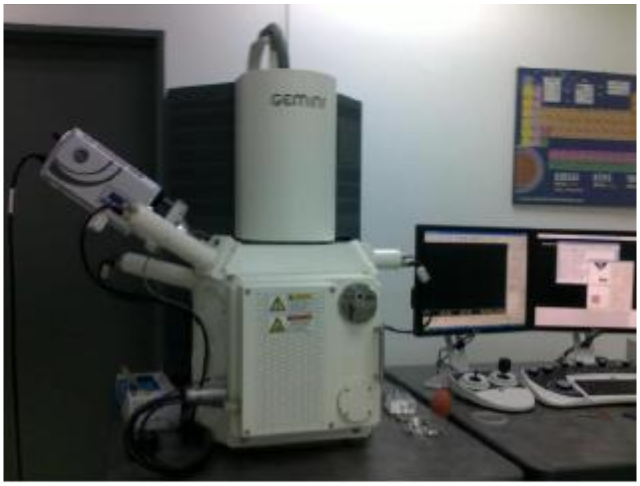

Figure 24 SEM

APPENDIX II

Calculation of 500 ppm of Zn(II) ion solution

In order to minimize error, the solution prepared is started from 500 ppm $ZnCl_{2;}$

$$100\ ppm\ by\ mass\ = \frac{500\ mg\ Zn(II)}{1\ kg\ of\ water}$$

Given the molar mass: Zn – 65.38 g/mol; $ZnCl_2$ – 136.28 g/mol

Assuming 1.0 mol of $ZnCl_2$, thus having 1.0 mol of Zn (II)

$$Mass\ of\ Zn(II)ions = \frac{65.38\ g\ Zn(II)}{1\ mol\ of\ Zn(II)} \times 1\ mol\ of\ Zn(II)$$

$$= 65.38\ g\ Zn(II)$$

$$Mass\ of\ ZnCl_2 = \frac{136.28\ g\ ZnCl_2}{1\ mol\ of\ ZnCl_2} \times 1\ mol\ of\ ZnCl_2$$

$$= 136.28\ g\ ZnCl_2$$

$$\therefore Percent\ mass\ of\ ion\ Zn(II) = \frac{65.38\ g\ Zn(II)}{136.28\ g\ ZnCl_2}$$

$$= \frac{0.4797\ g\ Zn(II)}{1.000\ g\ ZnCl_2}$$

Then, 500 mg of Zn(II) in $ZnCl_2$;

$$500 \times 10^{-3} g\ of\ Zn(II) \times \frac{1.000\ g\ ZnCl_2}{0.4797\ g\ Zn(II)} = 1.042\ g\ ZnCl_2$$

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

$$500V_1 = 100(1000ml)$$

$$V_1 = 200ml$$

Therefore, **200 ml of 500 ppm of Zn(II) ion solution** is diluted to prepare 25 ppm of Zn(II).

APPENDIX III

Calculation for Percent Metal Uptake

The calculation for metal uptake is such as below:

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

Calculation for Adsorption Capacity

The calculation for adsorption capacity is such as below:

$$\boxed{\frac{\text{Initial concentration} - \text{Final concentration}}{\text{Weight of rice husks in gram (g)}} \times \text{Volume of solution in liter (L)}}$$