Removal of Heavy Metal Ions from Industrial Wastewater Using Activated Carbon Derived from Rubber Seed Shell

by

Nur Atikah Binti Haji Abdullah
16939

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

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Universiti Teknologi PETRONAS
32610 Bandar Seri Iskandar
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

(Azry B. Borhan)

UNIVERITI TEKNOLOGI PETRONAS

SERI ISKANDAR, PERAK

May 2015

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.

NUR ATIKAH BINTI HAJI ABDULLAH

ABSTRACT

Rubber plantation is the second largest plantation in Malaysia where produced a large amount of rubber seed shell as a waste. To prevent odour pollution caused by the waste, a research was conducted to produce an activated carbon. The raw rubber seed shell has high carbon content about 52.5% which fall between ranges to be a prospective activated carbon. Heavy metal ions exist in almost industries nowadays and using adsorption method, activated carbon derived from rubber seed shell is used to remove the heavy metals especially in electroplating industry. Electroplating industry is one of major contributing industry producing higher amount of heavy metal even though undergo wastewater treatment process. Electroplating industry produces a high percentage of effluent zinc and copper. The rubber seed shell was grinded and sieved into two different size which are 1mm and 500µm. The rubber seed shell then further heated using oven, furnace and other equipment using activation temperature of 500˚C and 600˚C and activation time of 90, 120, 150, 180 and 210 minutes. Sample B1 is the best activated carbon produced using activation temperature of 600˚C, activation time of 180 minutes using impregnation ratio 1:2 with activating agent sodium hydroxide, NaOH solution. The removal of heavy metals of zinc and copper from electroplating industry was investigated based on three (3) effects of initial concentration, effect of stirring rate and effect of contact time. With specific surface area of $28.17\mathrm{m}^2\mathrm{g}^{-1}$ and pore diameter of 17.3nm, sample B1 removed zinc better than copper up to 99% of removal. From effect of initial concentration, sample B1 removed zinc up to 98.8% and copper up to 85%. For effect of stirring rate, percentage of zinc removal is 99.7% and copper is 99.6%. for effect of contact time, percentage of zinc removal is 99.6% and copper is 88%.

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LIST OF ABBREVIATION AND NOMENCLATURES

SMR	Standard Malaysia Rubber
FESEM	Field Emission Scanning Electron Microscopy
RSS	Rubber Seed Shell
AAS	Atomic Absorption Spectrophotometer
AC	Activated carbon

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CHAPTER 1

INTRODUCTION

1.1 Background

Heavy metals ions present in the industrial waste nowadays get many attentions from a lot of researchers to apply their study on removal of heavy metals using many ways such as by using the activated carbon derived from the agricultural waste. For example, in Malaysia, low-cost agriculture waste have been used is banana peel [1]. Heavy metal refers to any metallic chemical element that has a relatively high density and very toxic or poisonous at low concentration [2] which can cause diseases like cancer to human and cause bad pollution to environmental. Many researchers came out with their effort of investigation on heavy metals removal using a low-cost agriculture wastes that have high of carbon content such as palm kernel [3], rubber seed shell [4], banana peel [1], coconut shell [5], doum seed coat [6], walnut shell [7] and many more.

Malaysia is the fifth ranking natural rubber production country in the world after China, the USA, Japan and India while at the same time; Malaysia is the biggest manufacturer of latex-based sub-sector which is gloves [8]. From the latest report, world ranking in natural rubber consumption is China, the world ranking in natural rubber production; Malaysia was in the fourth place after, Thailand, Indonesia and Vietnam while the world ranking in rubber plantation, Malaysia was the third place after Indonesia and Thailand [9].

Rubber was known to the indigenous peoples of the Americas long before the arrival of European explorers in 1525 where Padre d'Anghieria reported that he had seenMexican tribespeople playing with elastic ball while the first research done on rubber was undertaken by Charles de la Condamine during his trip to Peru 1735 following with Fresnau investigation on a French engineer's home ground found that rubber is a "type of condensed resinous oil" [10].

Besides, Malaysian Rubber Board website stated that Natural Rubber (NR) was being introduced in Malaysia since 1877 when Sir Henry Wickham brought 22 seedlings from the batch of 70, 000 seedlings collected from Amazon. First location where the *Hevea brasiliensis* seedlings were initially planted is in the Residency gardens at Kuala Kangsar [11] and lead Malaysia to become the largest country exported the raw natural rubber in 80's. Natural rubber was divided into nine types of grade of the raw rubber materials in Malaysia based on [12] as per Table 1 below:

TABLE 1. Grade of Raw Rubber Materials in Malaysia [12]

Grade	1) SMR CV 50
	2) SMR CV 60
	3) SMR L
	4) SMR 5
	5) SMR GP
	6) SMR 10 CV
	7) SMR 10
	8) SMR 20 CV
	9) SMR 20

Nowadays, synthetic rubber becomes a challenge for natural rubber since the production of natural rubber cannot fulfil the requirement from the industries. The natural rubber cost is more expensive (SMR 20 = RM514.50) [13] than synthetic rubber, many industries decided to use synthetic rubber in order to produce more rubber less quality of rubber product. Table 2 below shows the differences of natural rubber and synthetic rubber.

TABLE 2. Natural Rubber versus Synthetic Rubber [14]

Characteristic	**Natural rubber (NR)**	**Synthetic rubber (SR)**
Source	NR is extracted from rubber trees, rubber grass and other plants and going through processing of gelatine	SR are made of various monomers after the polymerization
Physical properties	• High elasticity • High plasticity • Good mechanical strength • Small loss of hysteresis • Low heat production at multiple deformations • Good flexing resistance • Good insulation performance	• High elastic polymer materials • Resistant to high or low temperature
Chemical Properties	• Strong chemical reaction because of unsaturated double bond • Light, heat, ozone, radiation and flexible deformation and copper, manganese and other metal can promote the aging of rubber • Stabilizer can keep NR sheeting for a long time. • Good alkali resistance	• Air tightness • Oil resistant

Rubber can produce a lot of items used in life such as adhesive tapes, balloons, dock fenders, dental dams, erasers, floor coverings and mats, rubber bands, rubber gloves, tires and everything that connected in our daily life [15]. Based on the Malaysian Natural Rubber Industry, they found that the rubber plantation was the second largest plantation in Malaysia after palm oil, but only the rubber tree milk used as a chief product [11].

In Malaysia, the production of Natural Rubber for 2014 is about 342, 287 tonnes over 1, 077.92 (‘000 ha) which is show a decreasing order starting from year 2011 over 1, 027.04 (‘000 ha) which produce 996, 210 tonnes [16]. From latest information of rubber on November 2014, the rubber production in Malaysia was recorded at 53, 040 tonnes where increase about 11% (5, 252 tonnes) from October 2014 [17]. The natural rubber production trending will produce a higher amount of rubber waste and rubber seed is one of the biomass types has been ignored and left as waste [18]. The wastes left over should be recycling for something beneficial to the people and environment. The production of rubber in 2014 is 801.6(‘000) tonnes, where the consumption is 825.1 (‘000) tonnes and the imports of rubber is 1,226(‘000) tonnes and the exports is 1,312.7(‘000) tonnes which is show a decreasing order than 2013 [19].

The main rubber plantation in Malaysia located at Segi Tiga Jengka di Pahang, Southeast of Johore, South of Kelantan, Lembah Rajang and Kuching Sarawak, Tawau, Lahad Datu, Tenom and Kudat at Sabah [20]. As the Figure 1 below, the rubber and palm oil plantation was the main plantation in Malaysia dominantly by many companies involved in researches of rubber, palm oil, and other plants. The grey colour was represent the rubber plantation around Malaysia while the line symbol represent the palm oil plantation.

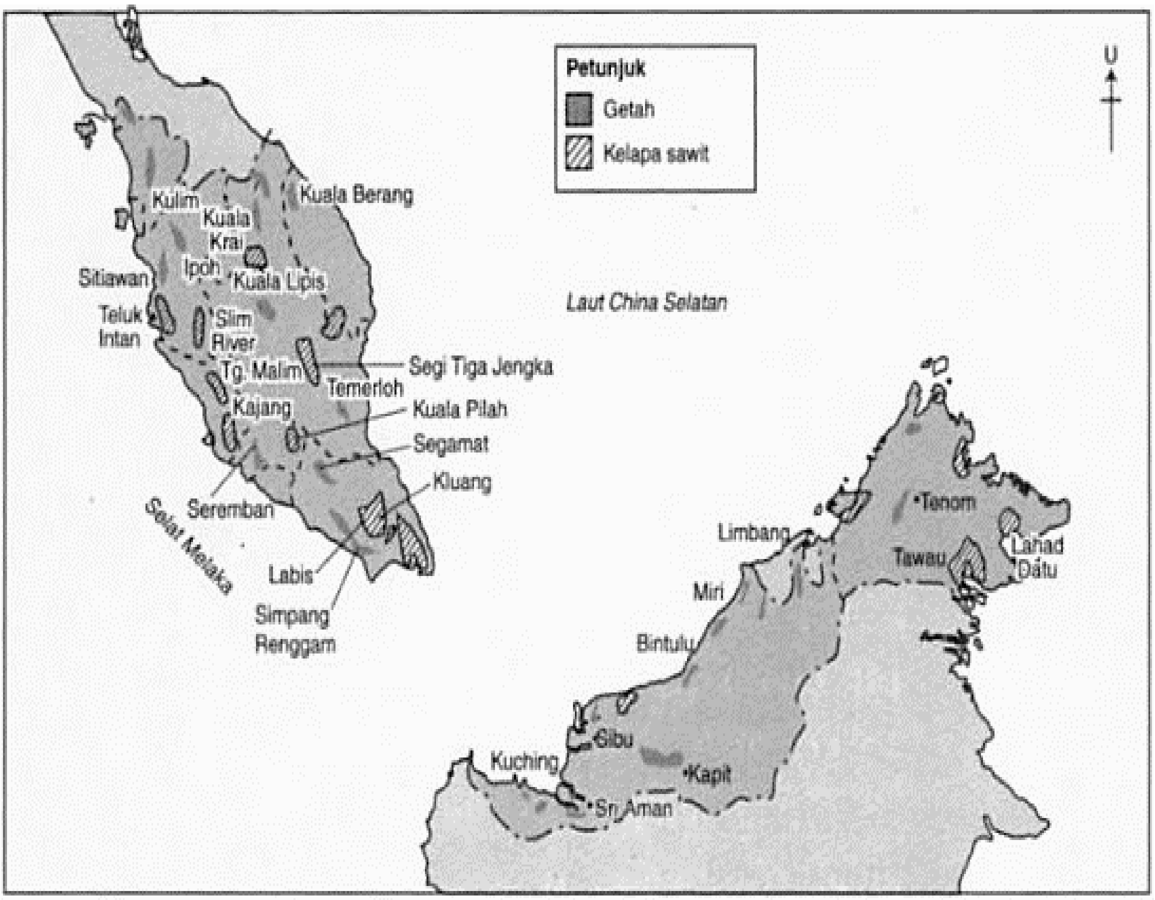


FIGURE 1. Map of Rubber Plantation in Malaysia [20]

Besides being an alternative to remove heavy metals in the industries, rubber seed shell also have another function in the construction industry since the high consumption can decrease the quantity of rubber seed shell unused as abundance in Malaysia. In this country, the effective in manufacturing concrete containing rubber seed shell would decrease the high dependency of concrete manufacturer towards natural collective and improve the application of this rubber seed shell as abundance waste in Malaysia [21].

Besides rubber seed shell, rubber seeds also a part of rubber plantation wastes that give a lot of advantages to industries. From the research done by Eka et al [22], well said that rubber seed can be a food, feed and also biofuel. As food, even though rubber seed contain low calcium and iron but foods that provide lower percentages of the Daily Values (DV) and Adequate Intake (AI) may also contribute to a healthful diet [22]. Rubber seed oil is similar to the common vegetable oils such as coconut oil, rubber seed meal and cakes are contain high protein supplement and amino acid contents of the rubber seeds indicate that bitter taste is more dominant [22]. Rubber seed is an important by-product of rubber plantation which has a high amount of semidrying oil used in paint industry, leaving a high-protein food for cattle or sheep, used a diet for broilers in Malaysia, for maize in poultry, and pig rations [22].

In addition, rubber tree also give advantages to the manufacturing industries to be applied as furniture and also essential in latex production which is extensively promoted and adjusted in industries for numerous years [23]. Rubber seed have great economic potential because from several researches reported that rubber seed contain 40-50% of oil shown its potential in oleo chemical applications in paints, surface coatings, soap, alternative to diesel and vulcanized vegetable oil production [23]. Besides, the rubber seed also comprise high protein and fats can be used in human food and animal feed as well as in production of soap, cosmetics and biofuel while in Jerantut, Pahang rubber seed has been fermented and used in curries and condiments [22].

Rubber seed oil (RSO) is one of the feedstock available in order to produce a higher amount of biodiesel. In Malaysia, there are a few private companies undergo biofuel production. According to Malaysia Biofuel Programme introduced the B5 blends of 95% petroleum diesel and 5% biofuels with cooperation of 5 big companies such as PETRONAS Trading Berhad, Shell Malaysia Trading Berhad, Esso Malaysia Berhad, Chevron Malaysia Berhad and Bousted Petroleum Marketing Sdn. Berhad with use of 113,000 tonnes of palm biodiesel in Central Region and another 37,000 tonnes in Southern Region in order to reflect the Malaysia’s commitment towards improving the energy security by substituting petroleum diesel with palm biodiesel [24]. Other countries which have major rubber plantation such as India also involved in biofuel production such as the Biodiesel Technologies Kolkata, India organized first Biodiesel Processing Plant with multi feedstock which are rubber seed oil, neem oil, waste vegetable oil, animal tallows and etc where can produced up to 3000L per day of biodiesel [25].

1.2 Problem Statement

Based on researches done by Wendy et al, rubber planting area of 1.02 Mha extracted 153t of rubber seed every year which it could be used as commercial substitute for biodiesel fuel [18]. According to the statistic of the natural rubber production every year, more than one million of the waste of the rubber seed shell produced also increase and it became abundance which lead to another problem which is land and odor pollution. Each rubber tree produces about 800 seeds (1.3kg) two times per annum and rubber plantation able to yield about 800 to 1200 kg rubber seed per ha per annum indirectly in 2007, 1,229,940 hectares of rubber plantation yields around 1000kg seeds per ha/yr, the projected annual production of rubber seeds in Malaysia would be 1.2 million metric tons. Besides, Malaysia trashes about 355,200,000 kg fat and 136,800,000 kg protein every annum since in the rubber seed kernel comprises 29.6% fat and 11.4% protein.

The rubber waste such as rubber seed shell was found contain high carbon and oxygen content by researchers and then they used as activated carbon used in industrial wastewater to remove heavy metal ions. Some of the industries in Malaysia, their wastewater treatment system was not able to remove 100% of metal ions such as mercury, cadmium, arsenic, zinc, chromium, lead and etc. Department of Environment (DOE) has set the limitation of the industry of oil and gas before dump the wastewater to the sea which is not exceeding 200mg/l [26].

Heavy metal ions actually exist in the industries such as textile industry, petroleum refining, mining activities, paint industry, pesticides and surely oil and gas industry. A huge amount of coloured wastewater containing dyestuffs and other organic compounds was produced by textile industry [27]. While, in other industries such as paints and pigments, glass production, mining operations, metal plating and battery manufacturing contain heavy metals likes Pb, Cu, Cd, Cr, Zn, Ni and Fe which can cause many diseases to human being and environmental [28].

For this project, we will focus on the electroplating industry because electroplating is a major polluting industry since it discharges toxic materials and heavy metals through wastewater (effluents), air emissions and solid wastes in environment [29]. While, the electroplating industry is the one of the industries produced 3% copper, 7% zinc, 0.1% chromium, 1% ferum, and 3% nickel [30]. Since 1995, the world production of copper increased by 65% [30].

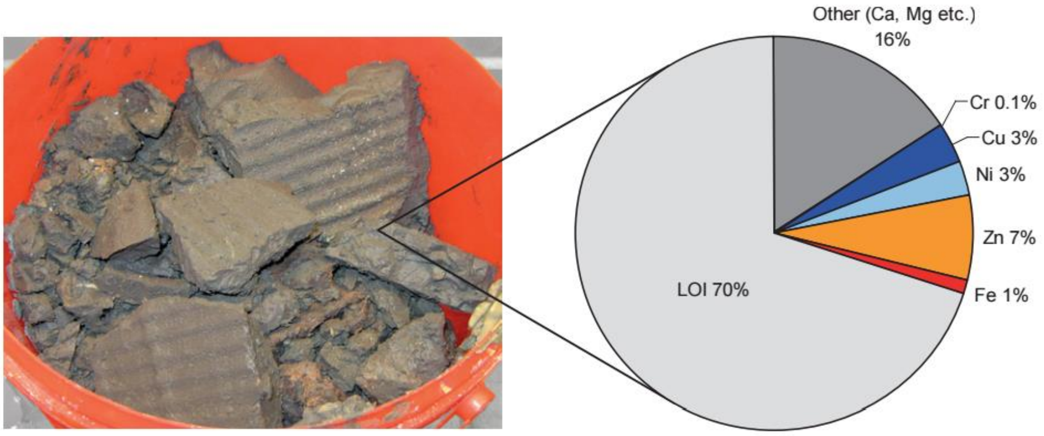


FIGURE 2. Sample of Electroplating Sludge Content Quantified By X-Ray Fluorescence Spectroscopy (LOI = Loss on Ignition)

Researches and investigation have been done on economy and obtainable agricultural wastes such as coconut shell, orange peel, rice husk, peanut husk and sawdust were used to be activated carbon and remove the heavy metal existing [28]. There are several techniques used in removal of heavy metal ions for instance distillation, precipitation, ion exchange, membrane filtration, electrolytic method, coagulation, reverse osmosis, and the most famous and versatile technique is the adsorption of heavy metal ion using activated carbon [31]. Heavy metals ions also can lead to the environment pollution directly can cause several diseases to living things. Heavy metals are any poisonous or toxic that has relatively high density at low concentrations which are natural components came from Earth's crust and they cannot be destroyed or degraded [32].

1.3 Objectives

1) Preparation and characterization of activated carbon using rubber seed shell by chemical activation using NaOH solution.
2) Investigate the effect on operating parameters such as activation temperature, activation time and impregnation ratio on the quality of activated carbon produced.
3) To test the performance of activated carbon produced in removing heavy metals ions such as Zinc and Copper from the electroplating industry wastewater.

1.4 Scope of Study

From the previous research, the rubber seed shell has the potential in removing the heavy metal ions such as silver, cadmium and copper [33]. This project will investigate the removal on two types of heavy metal ions which are Zinc (Zn) and Copper (Cu) using the selected activated carbon produced. The parameters that will be used in production of activation carbon are activation time and activation temperature while the factors affecting to heavy metals removal are:

1. Stirring rate
2. Initial concentration
3. Contact time.

CHAPTER 2

LITERATURE REVIEW AND/OR THEORY

2.1 Activated Carbon

Activated carbon is defined as a solid, adsorbent, carbonaceous substantial organised by carbonizing and activating carbon-based materials [34]. Activated carbon is one of the catalysts that have been used in the removal of metals ion in industry. Activated carbon also known as activated charcoal, activated coal or carbon activates [35] where the primary raw material is any organic material with high carbon content used for activated carbon [36]. An experiment was conducted on activated carbon from rubber seed shell and shows the element composition of raw rubber seed shell as in tables below [35]:

TABLE 3. Elemental Composition of Raw Rubber Seed Shell [35]

Element	Weight %	Atomic %
Carbon, C	52.50	59.55
Oxygen, O	47.50	40.45
Total	100.00	

TABLE 4. Element Composition of Activated Carbon (500˚C, 30mins) [35]

Element	**Weight %**	**Atomic %**
Carbon, C	80.70	84.78
Oxygen, O	19.30	15.22
Total	100.00	

As the Table 3 and Table 4 show the carbon content of the rubber seed shell is high thus, this kind of raw material is suitable to become the activated carbon in heavy metal removal. The carbon content of raw RSS is 52.50% (weight %) indicated that RSS is able to be a prospective activated carbon since its carbon content falls within the desirable range (50% - 80%) [37].

The activated is a porous material and “adsorption” process was taken place to eliminate any organic compounds from liquids and gases [35]. They define the activated carbon is the material that has an incredible capacity of adsorption, an attraction for a wide several of dissolved organics and chlorine and capability to be custom-tailored to suit precise applications. The activated carbon is black in coloured and the good production of activated carbon will be affected by several factors such as pH, agitation time, metal ion concentration, adsorbent dose, contact time, stirring rate and etc [31]. Besides become the activated carbon, rubber seed shell also have higher capability as biomass where other researcher have found several elemental composition exist in the rubber seed shell where it is very useful in biofuel production. Table 5 below shows the elemental analysis of rubber seed kernel with other biomass products.

TABLE 5. Elemental Composition of Malaysian Biomass [38]

Biomass	**Ultimate Analysis (daf, wt%)**				
	C	**H**	**N**	**S**	**O(by diff)**
Empty Fruit Bunch	40.73	5.75	1.40	0.22	92.63
Palm Kernel Shell	49.65	6.13	0.41	0.48	92.98
Rubber Seed Kernel	**44.01**	**6.11**	**0.58**	**0.03**	**93.28**
Sawdust	43.68	6.65	0.23	0.04	93.08
Coconut Fibre	45.51	6.02	0.78	0.09	93.11
Coconut Shell	43.00	6.30	0.75	0.05	92.90

From Table 5, it is approved that rubber seed kernel also have high carbon content thus, it proved that rubber plantation also can provide a low cost activated carbon used in the industries nowadays.

2.2 Adsorption

Adsorption is the main process happen where the pores of the activated surface area will be opened and attract the metal ions. The higher number of pores on the activated carbon will affect the adsorption rate of the metal ions. Adsorption defined as the process by which molecules of gases or liquids will be attached at the surface of another substance or adsorbent. Desorption is the opposite process. The surface of the adsorbent is known as the adsorbate [1].

Adsorption was divided into two categories, namely physisorption and chemisorption. Physisorption normally comprises weaker bonding forces among the molecules (e.g. Van Der Waals force) which is typically happens between the adsorbed molecules and the adsorbent internal pore surface. Another characteristic of physisorption is desorption can be accomplished easily because it is readily reversible. While for chemisorption basically involves strong chemical bonding (e.g. covalent bond) cause more selective and highly specific process on the adsorbate molecules and it usually occurs in monolayer [1].

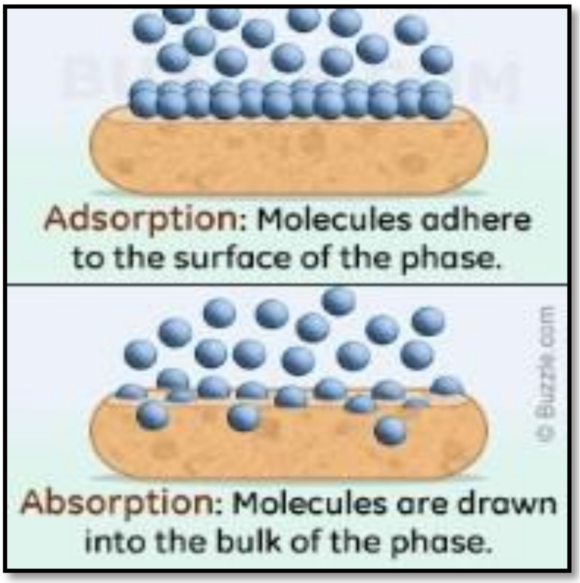


FIGURE 3. An Overview Diagram on Difference between Adsorption and Absorption

2.3 Chemical and Physical Activation

Activated carbon of the raw material will be prepared using two different method which are chemical activation by using any chemical reagent and the other method is activate the raw material by physical activation. Both methods will use gas flow in order to transform the raw material into activated coal which is black in colour [39].

Physical activation encompasses of the raw material carbonization and then following with the activation at high temperature in a carbon dioxide or steam atmosphere while the chemical activation method comprises of the raw material impregnated with chemical reagent such as NaOH, KOH, $ZnCl_2$, H_3PO_4, HCl, H_2SO_4, CH_3COOH and etc. before being carbonized under high temperature [39].

FIGURE 4. Example of Activated Carbon

Based on Table 6, using sodium hydroxide solution can produced higher surface area and pore volume formation of activated carbon rather than using potassium hydroxide as activating agent. By varying the ratio activating agent and raw material from 2:1, 4:1, 6:1 and 8:1, the higher the amount of activating agent increase the surface area and pore volume of the activated carbon produced [40]. The higher the specific surface area of the activated carbon, the higher the performance of the adsorption.

TABLE 6. Difference Surface Area and Pore Volume Using Activating Agent KOH and Naoh Solution

Activ. agent	**Ratio (wt:wt)**	**Act. Temp (°C)**	**Act. Time (h)**	**Yield (%)**	**Total pore volume, $V_T (m^3 g^{-1})$**	**Specific surface area S_{BET}, $(m^2 g^{-1})$**
KOH	2:1	750	1	78	0.49	1090
KOH	4:1	750	1	67	0.78	1635
KOH	6:1	750	1	60	0.90	2225
KOH	8:1	750	1	47	0.94	2420
NaOH	2:1	750	1	71	0.51	1130
NaOH	4:1	750	1	55	0.81	2000
NaOH	6:1	750	1	37	0.96	2541
NaOH	8:1	750	1	27	1.02	3033
CO_2		890	3	80	0.29	644
CO_2		890	9	45	0.64	1528
CO_2		890	22.5	6	0.86	2487

Each of the method of activation of activated carbon has their own advantages and disadvantages. For chemical activation, one of the advantages is activated carbon produced using this method basically has higher surface area because of the existent of activating agent like sodium hydroxide and potassium hydroxide. Table 7 and Table 8 discuss about the advantages and disadvantages of physical and chemical activation.

TABLE 7. Advantages of Physical and Chemical Activation [41]

Physical Activation	**Chemical Activation**
✓ Physical activation produce a “clean” activated carbon without combination any inorganic scums ✓ Produce significant microporous carbon which is beneficial for gaseous effluent applications	✓ Can yield activated carbon with a large pore distribution at low temperature (<500˚C). ✓ Chemical reagent of phosphoric acid is favoured because it contributes slight impact to the environment and after carbonization it can be recycle/recover from washing step. ✓ A short activation time ✓ Higher BET surface area (1800-2200 m^2/g) ✓ Nitrogen and air increase micropores volume of activated carbon ✓ Steam increase mesoporosity and microporosity. ✓ Gaseous atmosphere (N_2 and O_2) produce acidic properties to the carbons.

TABLE 8. Disadvantages of Physical and Chemical Activation [41]

Physical Activation	**Chemical Activation**
✓ From the first step of carbonization process, it will remove volatile materials such as hydrogen and oxygen because of the high temperature heating. The "combustion" process formed tar and hydrocarbons such as methane and methanol which eliminate a big quantity of available carbon. ✓ Second step, consume higher temperature (900˚C-1000˚C) ✓ Extended activation time ✓ Weak yield of activated carbon (15%) to get surface area BET 1100m^2/g ✓ Small pore distribution	✓ Produce impurities likes Zn and P, according to the chemical reagent used. ✓ Pore size distribution and surface chemistry play a significant part on adsorption phenomenon of gas, liquids and solids thus, make sure use or buy adequate amount of activated carbon for the application. ✓ Chemical reagents such as NaOH, KOH, $ZnCl_2$,NH_4Cl, $BaCl_2$, $MgCl_2$, K_2CO_3, K_2HPO_4, and H_3PO_4 expensive than CO_2 and H_2O used in physical activation.

2.4 Rubber Seed Shell

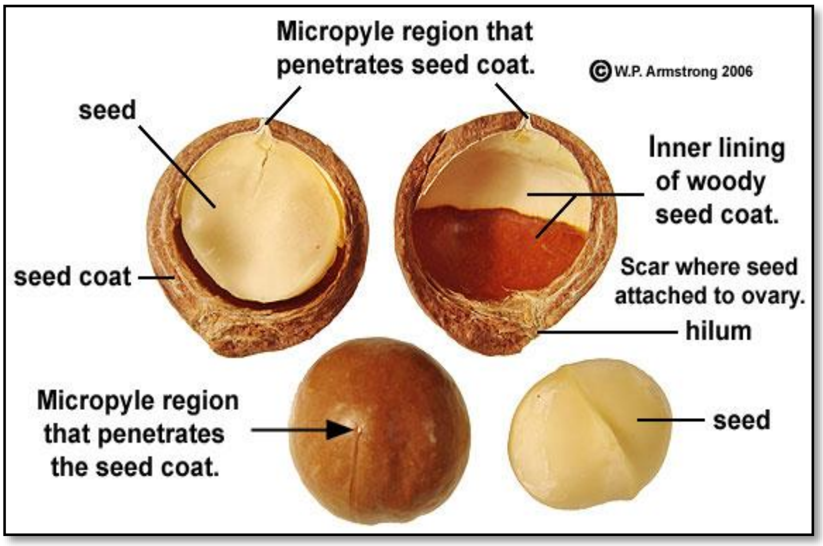


FIGURE 5. The Natural Rubber Characteristic

Natural rubber characteristic contain of seed, seed coat, micropyle region that penetrates seed coat, hilum and inner lining of woody seed coat. The Malaysian natural rubber, *Heavea brasiliensis* have a potential use as food for human, feed for animals and biofuel for energy [22]. The reprocess of solid waste from rubber seed shell give profits to environment in treating heavy metals in wastewater and the objective of this research is to assess the probability of implementing rubber seed shell as adsorbents for the adsorption of heavy metals in wastewater by applying the deviation of certain parameters [33]. Rubber seeds contain 43% oil while every 100g seeds contain 8.5g water, 17.6g protein, 48.5g fat, 22.9g total carbohydrate, 2.5g ash, 120mg calcium and 430mg phosphorus suitable as biofertilizer by pot culture method [42].

FIGURE 6. Outer Cover of Rubber Seed Shell and Rubber Seed Shell

Beneath rubber agricultural estate with 1.3 million ha of area varying from more than 20 clones of rubber trees and each hectare will provide an estimated quantity of 150kg of seeds which have been establish to be rich in oil and were not edible but could be found in abundance in country, thus the production of biofuel grab the consideration and shows the growing in both quantity and quality in Malaysia [43].

In this article also mentioned about the function of raw material of rubber seed shell as an activated carbon production while the rubber seed oil functioning in biodiesel production were investigated by various study and one of them is used in kaolin intercalates [43].

FIGURE 7. Rubber Seed Shell Waste

The figure 7 above is the picture of waste produced from the natural rubber. This rubber shell waste contains higher amount of carbon and oxygen which convert the cheap and available agricultural product to useful and high-value adsorbents and it can be used in organic chemicals and metals removal [44]. Since the production of natural rubber every year reached half million tonnes, the abandoned waste will be increasingly without any requirement needed to do with them. The scientists found from the abandoned waste like rubber seed shell have higher carbon content and it is proven when derived as an activated carbon. Besides become activated carbon to remove heavy metal ions from the wastewater industries, rubber seed shell also can be produce as biofuel for energy.

2.5 Heavy Metal Ions

Many industries such as fuel and power manufacturing produce 2.4 million tons of As, Cd, Cr, Cu, Hg, Ni, Pb, Se, V and Zn every year. The metal industry produces 0.39 million tons/year of the similar metals to the environment, while agriculture industry contributes 1.4 million tons/year, manufacturing ads 0.24 million tons/year and waste disposal complements 0.72 million tons/year. Metals, discharged or transported into the environment, may endure alterations and can have a huge environmental, public health, and economic effect [45]. The pollutants comprise of cadmium, lead, mercury, chromium, arsenic, zinc, cobalt, copper and nickel have a quantity of applications in simple industrial works, paper and pulp industries, leather tanning, petrochemicals, fertilizers and so on which each of them bring bad effect on human health [45].

Department of Environment (DOE) from Malaysia have extracted the rules and regulations from Environmental Quality (Industrial Effluents) Regulations 2009 to every industry where they should comply the acceptable conditions for discharge of industrial effluent for mixed effluent of standard A and B [26]. Table 9 and Table 10 below show the acceptable condition should be comply by any industries before release the effluent to the sea.

TABLE 9. Acceptable Conditions for Discharge of Industrial Effluent

ACCEPTABLE CONDITIONS FOR DISCHARGE OF INDUSTRIAL EFFLUENT FOR MIXED EFFLUENT OF STANDARDS A AND B				
	Parameter	**Unit**	**Standard**	
			A	**B**
	(1)	**(2)**	**(3)**	**(4)**
i	Temperature	˚C	40	40
ii	pH value	-	6.0-9.0	5.5-9.0
iii	BOD at 20˚C	mg/L	20	40
iv	Suspended solids	mg/L	50	100
v	Mercury	mg/L	0.005	0.05
vi	Cadmium	mg/L	0.01	0.02
vii	Chromium, Hexavalent	mg/L	0.05	0.05
viii	Chromium, Trivalent	mg/L	0.20	1.0
ix	Arsenic	mg/L	0.05	0.10
x	Cyanide	mg/L	0.05	0.10
xi	Lead	mg/L	0.10	0.5
xii	Copper	mg/L	0.20	1.0
xiii	Manganese	mg/L	0.20	1.0
xiv	Nickel	mg/L	0.20	1.0
xv	Tin	mg/L	0.20	1.0
xvi	Zinc	mg/L	2.0	2.0
xvii	Boron	mg/L	1.0	4.0
xviii	Iron (Fe)	mg/L	1.0	5.0
xix	Silver	mg/L	0.1	1.0
xx	Aluminium	mg/L	10	15
xxi	Selenium	mg/L	0.02	0.5
xxii	Barium	mg/L	1.0	2.0
xxiii	Fluoride	mg/L	2.0	5.0
xxiv	Formaldehyde	mg/L	1.0	2.0
xxv	Phenol	mg/L	0.0001	1.0
xxvi	Free Chloride	mg/L	1.0	2.0
xxvii	Sulphide	mg/L	0.50	0.50
xxviii	Oil and Grease	mg/L	1.0	10
xxix	Ammoniacal Nitrogen	mg/L	10	20
xxx	Colour	ADMI*	100	100

ADMI – American Dye Manufactures Institute

TABLE 10. Acceptable Conditions for Discharge of Industrial Effluent (COD Contain)

Extracted from Environmental Quality (Industrial Effluents) Regulations 2009 (PU(A)-434)

SEVENTH SCHEDULE
(Regulation 12)
ACCEPTABLE CONDITIONS FOR DISCHARGE OF INDUSTRIAL EFFLUENT CONTAINING CHEMICAL OXYGEN DEMAND (COD) FOR SPECIFIC TRADE OR INDUSTRY SECTOR

(1) **Trade /Industry**	(2) **Unit**	(3) **Standard**	(4) **Standard**
(a) Pulp and paper industry			
i. Pulp mill	mg/L	80	350
ii. Paper mill (recycled)	mg/L	80	250
iii. Pulp and paper mill	mg/L	80	300
(b) Textile Industry	mg/L	80	250
(c) Fermentation and distillery industry	mg/L	400	400
(d) Other industries	mg/L	80	200

EIGHT SCHEDULE
(Regulation 13)
ACCEPTABLE CONDITIONS FOR DISCHARGE OF MIXED EFFLUENT CONTAINING CHEMICAL OXYGEN DEMAND (COD)

(1) **Unit**	(2) **Standard A**	(3) **Standard B**
mg/L	80	200

Any industries did not comply with this regulation will be fined up to a maximum of RM2,000.00 or offenders can be accused in court and the supreme penalty is RM50,000.00 or imprisonment for a period not more than 2 years or both and to further fine not more than RM1000.00 per day for every day the fault is sustained [26].

CHAPTER 3

METHODOLOGY

The flow of the project discovered based on experimental laboratory work. The result obtained from the experimental basically an empirical result that can be compared with the other research on removal of heavy metal ions. The activated carbon derived from rubber seed shell will be the adsorbent in the wastewater treatment system to remove the chosen heavy metal ions from the electroplating industry which are Copper, Cu (II) and Zinc, Zn (II). After the best sample of activated carbon have determined, then the adsorption experimental on heavy metal removal will be conducted. The experiment is divided into four main steps which are the first step is the rubber seed shell preparation of rubber seed shell, followed by second step of the characterization and activation of activated carbon. Then, the third step is the sample characterization where to determine the best sample of activated carbon produced. After the best activated carbon was determined, the experiment proceed to the removal of heavy metal ions based on three different affecting factors which are effect of initial concentration, effect of stirring rate and effect of contact time on percentage of heavy metal removal.

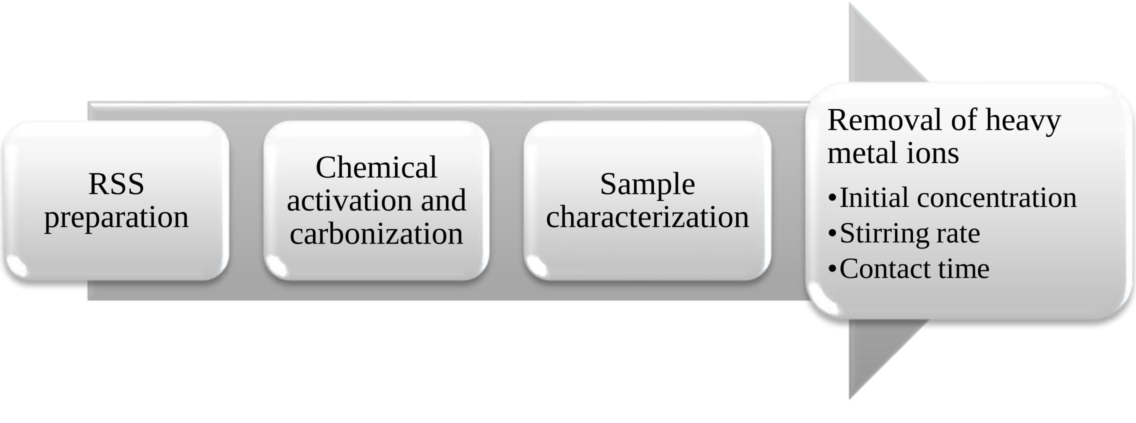
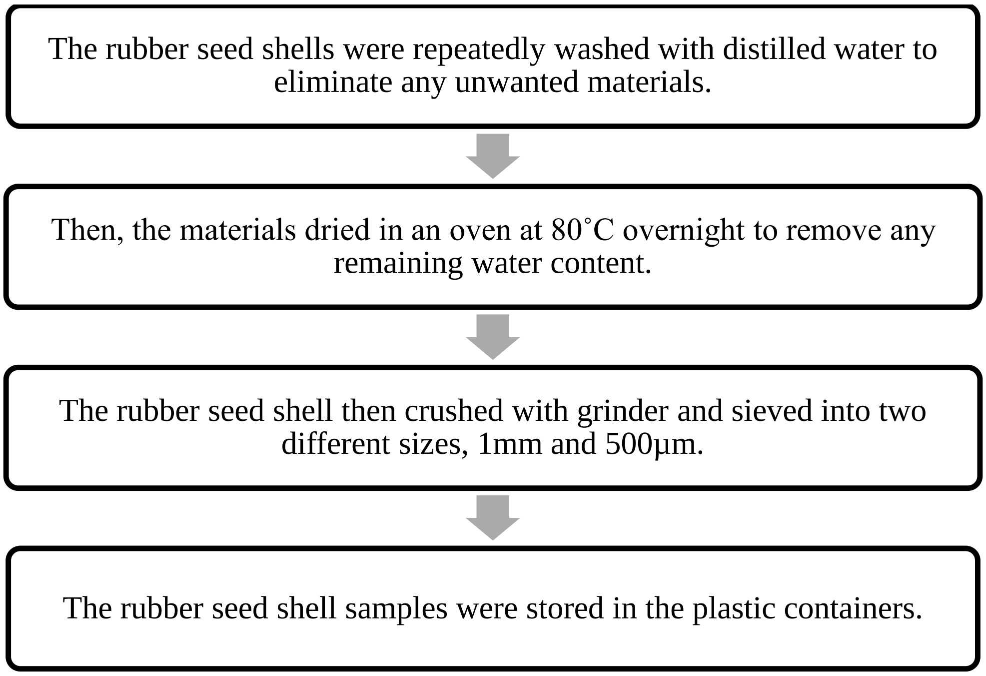


FIGURE 8. Flowchart of Experiment Plan

3.1 Rubber Seed Shell Preparation



The rubber seed shells were repeatedly washed with distilled water to eliminate any unwanted materials.

Then, the materials dried in an oven at 80˚C overnight to remove any remaining water content.

The rubber seed shell then crushed with grinder and sieved into two different sizes, 1mm and 500µm.

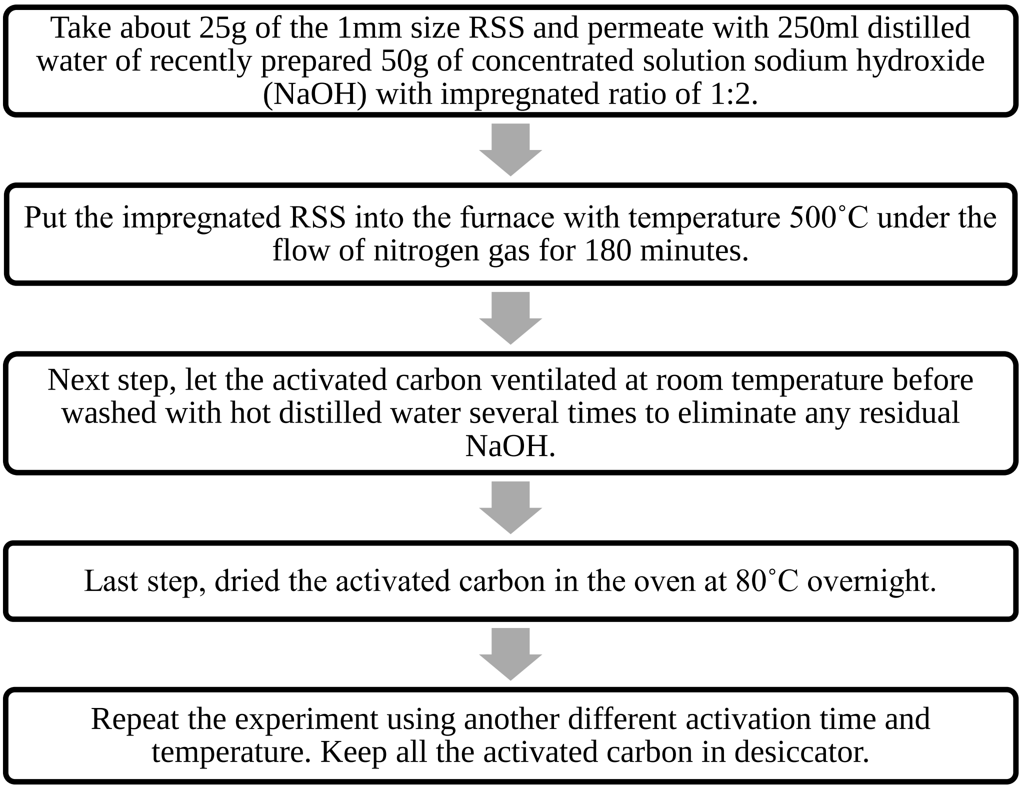
The rubber seed shell samples were stored in the plastic containers.

FIGURE 9. Flowchart of RSS Preparation

Rubber seed shell (RSS) is collected from RISDA farm located at Bota Kanan, Perak. The RSS were repeatedly washed with distilled water to eliminate any unwanted materials like sand and dirt existing in it. Then, the RSS sample dried in an oven at 80˚C overnight to remove any remaining water content in the RSS materials. The RSS then crushed with grinder and sieved into two different sizes which are 1mm and 500µm. The RSS sample were stored in the plastic container and keep in the desiccator.

FIGURE 10. Grinder and Siever Used to Crush RSS Sample

3.2 Chemical Activation and Carbonization



Take about 25g of the 1mm size RSS and permeate with 250ml distilled water of recently prepared 50g of concentrated solution sodium hydroxide (NaOH) with impregnated ratio of 1:2.

Put the impregnated RSS into the furnace with temperature 500˚C under the flow of nitrogen gas for 180 minutes.

Next step, let the activated carbon ventilated at room temperature before washed with hot distilled water several times to eliminate any residual NaOH.

Last step, dried the activated carbon in the oven at 80˚C overnight.

Repeat the experiment using another different activation time and temperature. Keep all the activated carbon in desiccator.

FIGURE 11. Flowchart of Chemical Activation and Carbonization

For activation and carbonization of RSS sample, the activating agent used is sodium hydroxide (NaOH) based on the previous research conducted where this chemical produce better pore size on the sample. The range of activation temperature are 500˚C and 600˚C while the activation time are 90, 120, 150, 180 and 210 minutes. The impregnation ratio used is constant as 1:2 (wt%).

$$\text{Impregnation ratio} = \frac{W_{RSS}}{W_{NaOH}} \tag{1}$$

50g of NaOH was prepared and dissolved with 250mL distilled water. Then, weight about 25g of RSS sample and permeated with NaOH solution for 24 hours thus the chemical substance will entirely adsorbed into the raw RSS.

FIGURE 12. RSS Sample Permeated With NaOH

Then, RSS sample was filtered and prepared for carbonization step. The impregnated RSS then put into a tube furnace with temperature 500˚C for 180 minutes. To activate the adsorbent, nitrogen gas will be used to purge the furnace at rate of $(100 cm^3/min$ and heating gradient at 10˚C/min for the scheduled time.

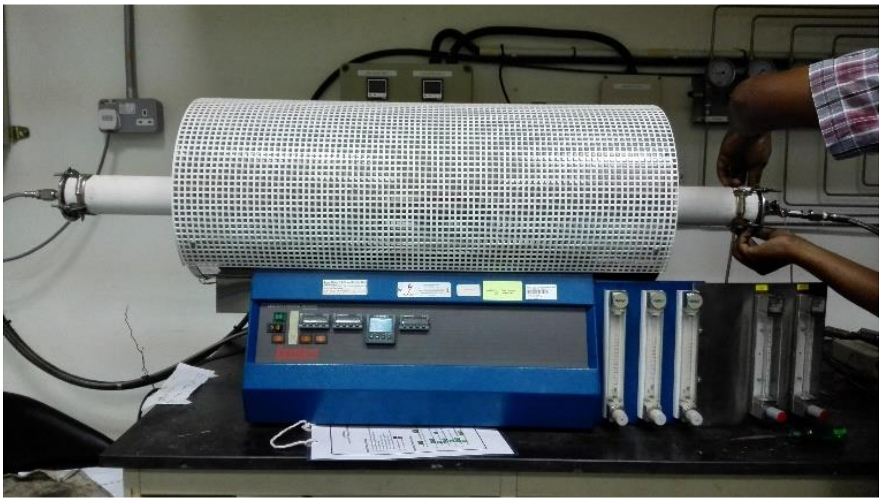


FIGURE 13. Tube Furnace Used for Activation and Carbonization

During the carbonization step, the impregnated RSS will goes into dry powder with black in color. Next step, let the activated carbon ventilated at room temperature before washed with 100˚C of distilled water several times to eliminate any residual NaOH. Lastly, dried the activated carbon in the oven at 80˚C overnight and keep in the desiccator.

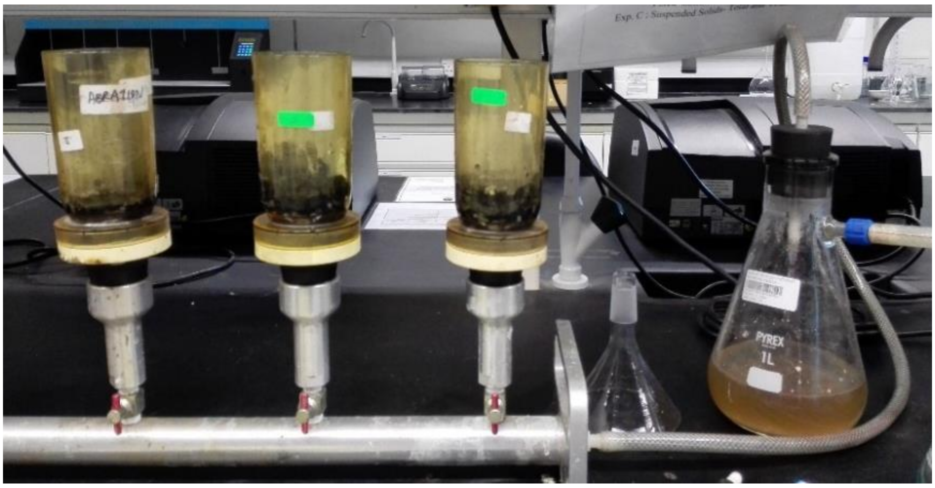


FIGURE 14. Vacuum Pump Used to Wash Activated Carbon with Hot Distilled Water

FIGURE 15. Activated Carbon Dried in Oven Overnight

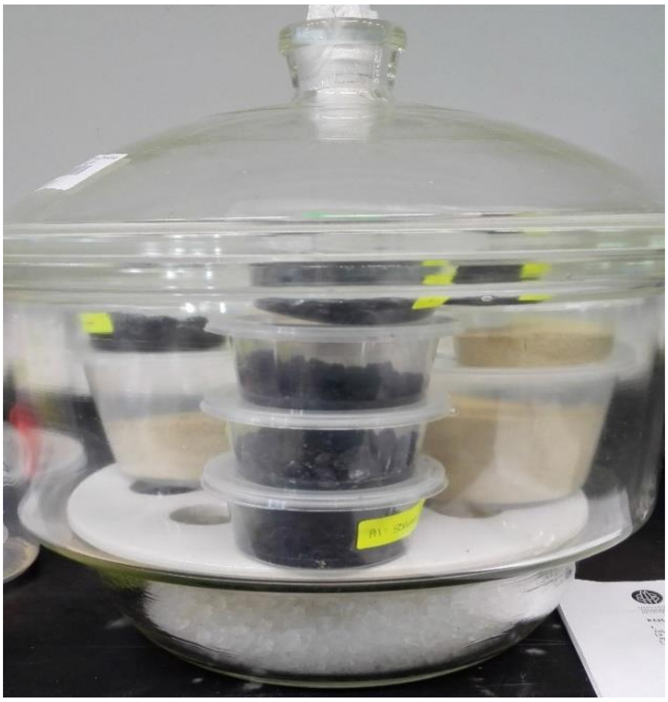


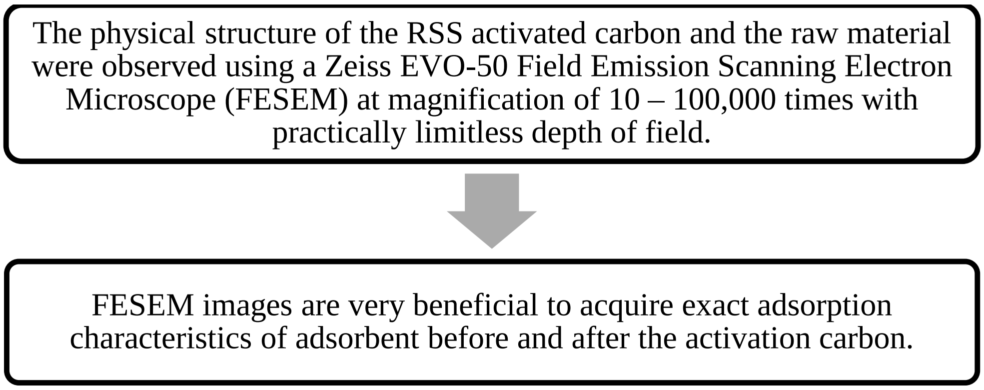
FIGURE 16. Desiccator Used to Keep the Activated Carbon Sample

The activation and carbonization steps were repeated using another different activation time and temperature. Table 11 below shows the activated carbon preparation of rubber seed shell for 1mm and 500µm.

TABLE 11. Preparation of Activated Carbon of RSS

Sample	Activation temperature (˚C)	Activation time (mins)
A	500	180
B	600	180
C	500	150
D	600	150
E	500	210
F	600	210
G	500	120
H	600	120
I	500	90
Raw	-	-

3.3 Sample Characterization



The physical structure of the RSS activated carbon and the raw material were observed using a Zeiss EVO-50 Field Emission Scanning Electron Microscope (FESEM) at magnification of 10 – 100,000 times with practically limitless depth of field.

FESEM images are very beneficial to acquire exact adsorption characteristics of adsorbent before and after the activation carbon.

FIGURE 17. Flowchart for Sample Characterization

The physical structure of the RSS activated carbon and the raw material were observed using a Zeiss EVO-50 Field Emission Scanning Electron Microscope (FESEM) at magnification of 10-100,000 times with practically limitless depth of field. FESEM images are very beneficial to acquire exact adsorption characteristics of adsorbent before and after the activation carbon.

FIGURE 18. Field Emission Scanning Electron Microscope (FESEM)

The pore size distribution, specific surface area and the pore volume of the activated carbon samples were determined by the nitrogen adsorption-desoprtion isotherm characterized by the Micromeritics ASAP 2020, by using nitrogen as the adsorbate while being degassed at 250˚C for five hours. The specific surface area of the activated carbon samples are estimated using the Brunauer-Emmett-Teller (BET) method using nitrogen gas adsorption isotherm data while the Barett-Joyner-Halenda (BJH) adsorption model is used to for the pore size distribution [46]

3.4 Factor Affecting the Removal of Heavy Metal Ions

The heavy metal solution used are Zinc Sulphate and Copper Sulphate. It is because as mentioned in the problem statement, the zinc and copper are the most contributor of heavy metal waste in the electroplating industry. The size of adsorbent used is 500µm only since the smaller the size, the higher the surface area then it will increase the rate of adsorption.

3.4.1 Effect of Initial Concentration

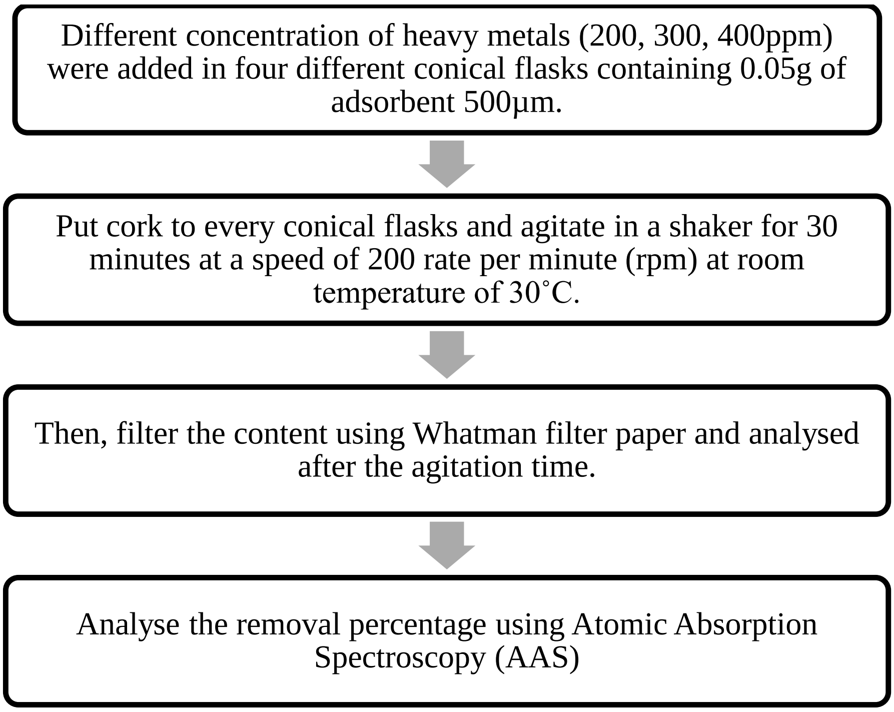


FIGURE 19. Flowchart of Initial Concentration Effect

Three different of zinc sulphate concentration range between 200ppm, 300ppm, and 400ppm are prepared and placed 100mL into four 250mL of conical flasks. Label the conical flasks with A1, B1 and raw sample. Then, approximately 0.05g of 500µm adsorbent are placed into the conical flasks. Put cork at every conical flasks and agitate on hot plate for 30 minutes at speed of 200 rate per minute (rpm) at room temperature of 30˚C. Then, the content of the solution are filtered using Whatman filter paper and analysed after the agitation time. The experiment were repeated using copper sulphate solution. The percentage of heavy metal removal were analysed using Atomic Absorption Spectrophotometer (AAS).

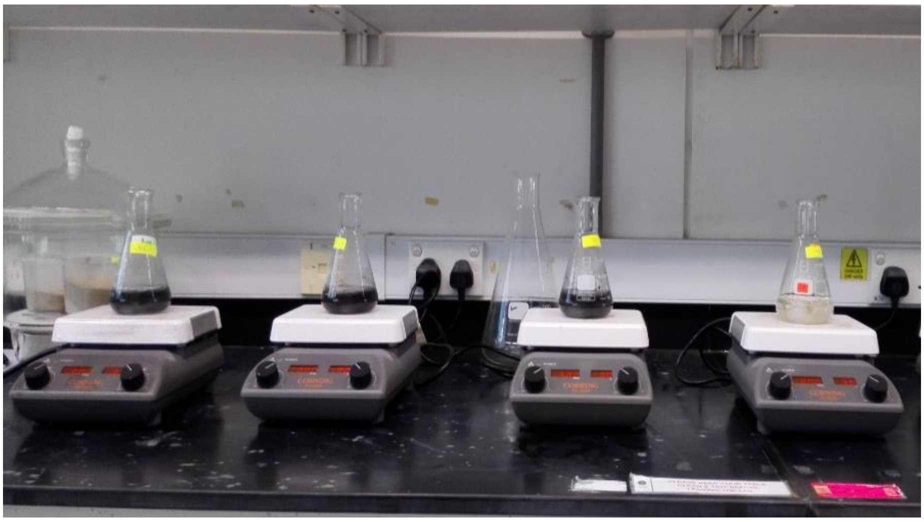
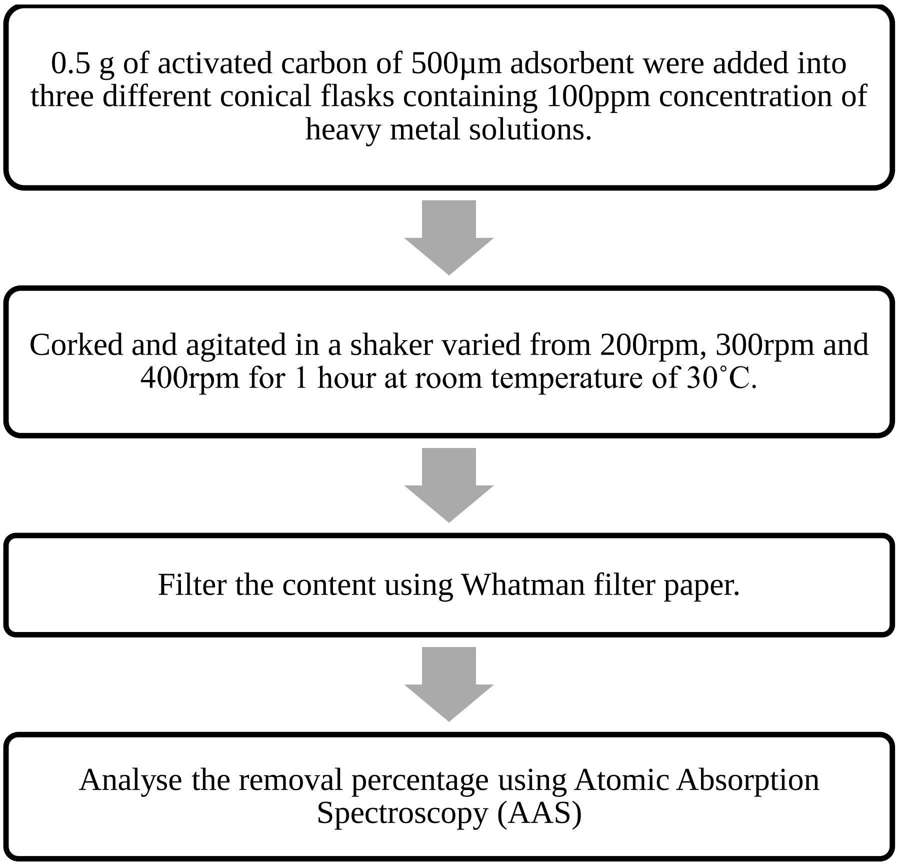


FIGURE 20. Experimental on Effect of Initial Concentration

FIGURE 21. Atomic Absorption Spectrophotometer (AAS)

3.4.2 Effect of Stirring Rate



0.5 g of activated carbon of 500µm adsorbent were added into three different conical flasks containing 100ppm concentration of heavy metal solutions.

Corked and agitated in a shaker varied from 200rpm, 300rpm and 400rpm for 1 hour at room temperature of 30˚C.

Filter the content using Whatman filter paper.

Analyse the removal percentage using Atomic Absorption Spectroscopy (AAS)

FIGURE 22. Flowchart of Stirring Rate Effect

A one litre of zinc sulphate with 100ppm concentration was prepared and placed 100mL into four different 250mL of conical flasks. Label the conical flasks with A1, B1 and raw sample. Approximately 0.5g of 500µm adsorbent was placed into each of conical flasks. Corked them and put on hot plates. Varied the stirring rate from 200rpm, 300rpm and 400rpm for an hour at room temperature of 30˚C. Then, filter the solution using filter paper. Repeat from the first step using copper sulphate solution. Analyse the percentage of removal using AAS.

3.4.3 Effect of Contact Time

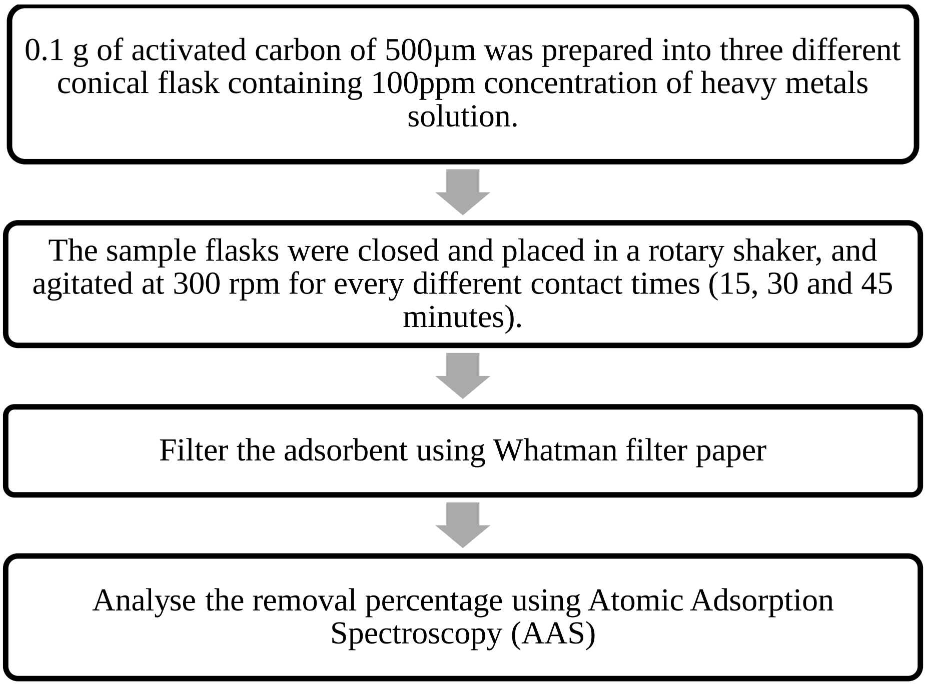


FIGURE 23. Flowchart of Contact Time Effect

A one litre of zinc sulphate solution with 100ppm concentration was prepared and placed 100mL into four different 250mL of conical flasks. The conical flasks were labelled with A1, B1, C1 and D1. Approximately 0.1g of 500µm of adsorbent were put into each of conical flasks. Corked then and placed on the hot plates. The experiment started when the constant stirring rate of 300rpm was set at 30˚C of room temperature and differentiate the contact times of 15, 30 and 45 minutes. Then, the solution were filtered using filter paper. The experiment was repeated from the first step using copper sulphate solution. The percentage of heavy metal removal were analysed using AAS.

3.5 Project Gantt Chart and Key Milestone

3.5.1 Gantt Chart and Key Milestone FYP 1

No.	Detail/Week	FYP I													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic	■	■												
2	Preliminary Research Work		■	■	■	■									
3	Submission of Extended Proposal						●								
4	Proposal Defence								■	■					
5	Project work continues										■	■	■		
6	Submission of Interim Draft Report													●	
7	Submission of Interim Report														●

■ Process ● Suggested Milestone

3.5.2 Gantt Chart and Key Milestone FYP II

No	Detail/Week	FYP II														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues	■	■	■	■	■	■	■								
2	Submission of Progress Report								●							
3	Project Work Continues								■	■	■	■	■			
4	Pre-SEDEX											●				
5	Submission of Draft Final Report												●			
6	Submission of Dissertation (Soft Bound)													●		
7	Submission of Technical Paper													●		
8	Oral Presentation														●	
9	Submission of Project Dissertation (Hard Bound)															●

■ Process ● Suggested Milestone

CHAPTER 4

RESULTS AND DISCUSSION

4.1 FESEM Image of Samples

The activated carbon sample of production can be determined using the formula for the sample yield calculation from [1] as stated below:

$$\text{Yield} = \frac{\mathrm{M_{AC}}}{\mathrm{M_{RSS}}} \times 100\% \qquad (2)$$

Whereby:

M_{AC}= dry mass of the activated carbon after washing (g)
M_{RSS}=original mass of the rubber seed shell powder before carbonization (g)

The activated carbon has an average yield of **49.02%.** The weight of the raw rubber seed shell was fixed at 15 to 20g but after several transfers from beaker into boat for activation and carbonization stage maybe there are some losses. Same goes during the washing and drying stage, there also cause some losses of the samples.

The following Figure 24, Figure 25, Figure 26 and Figure 27 are the activated carbon samples of A1, A2, B1 and D1. This samples were chose because from Table 12, sample A1 and B1 show the highest specific area formed. While the raw sample was chosen because to investigate the existence of pore size on the raw RSS.

The adsorption of the chemical activating agent is for the pore activation development. Based on the FESEM imaging, at 10-1000 times magnification on the samples, sample B1, A1 (500µm) and A2 (1mm) show the pores formation after activation and carbonization. While for raw sample, there are a few formation of pores on the surface of raw rubber seed shell. The images collected only the raw sample and the best activation and carbonization from previous research.

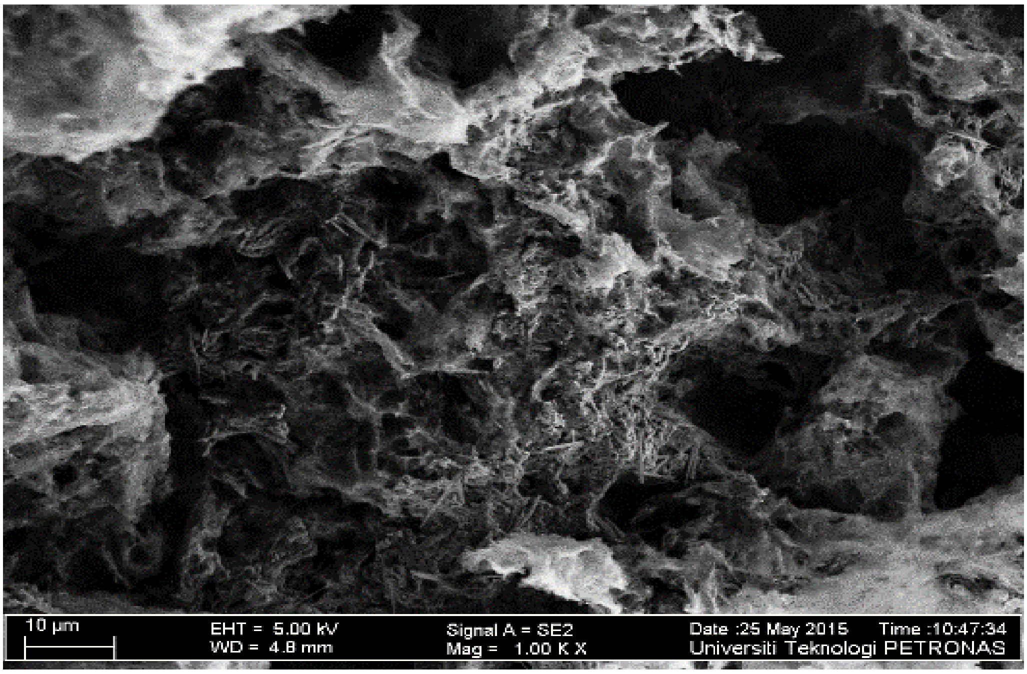


FIGURE 24. Sample A1

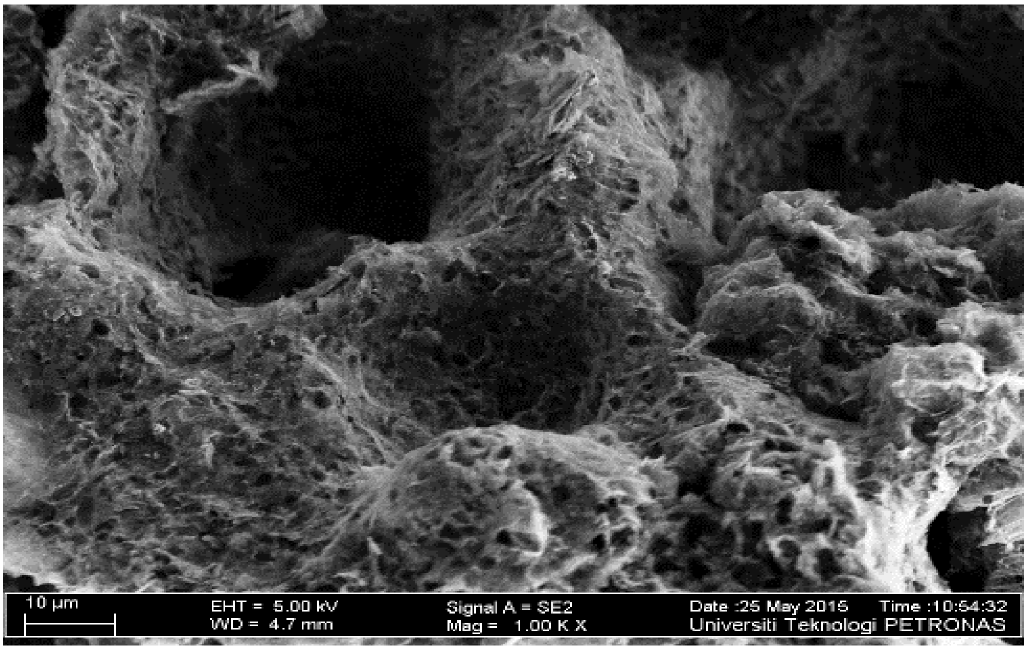


FIGURE 25. Sample A2

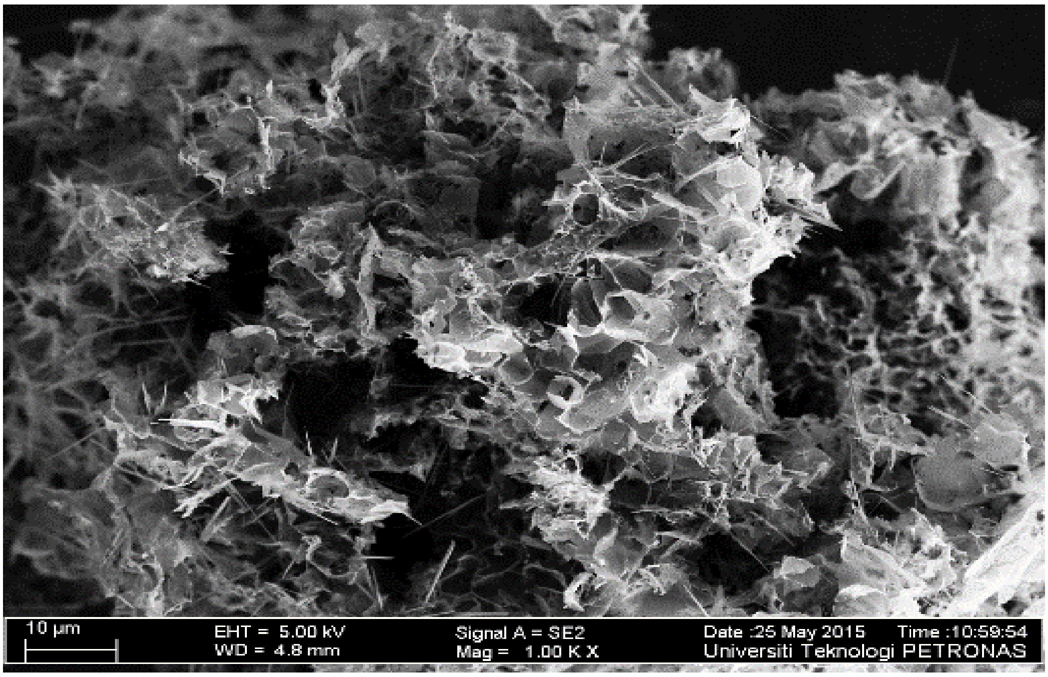


FIGURE 26. Sample B1

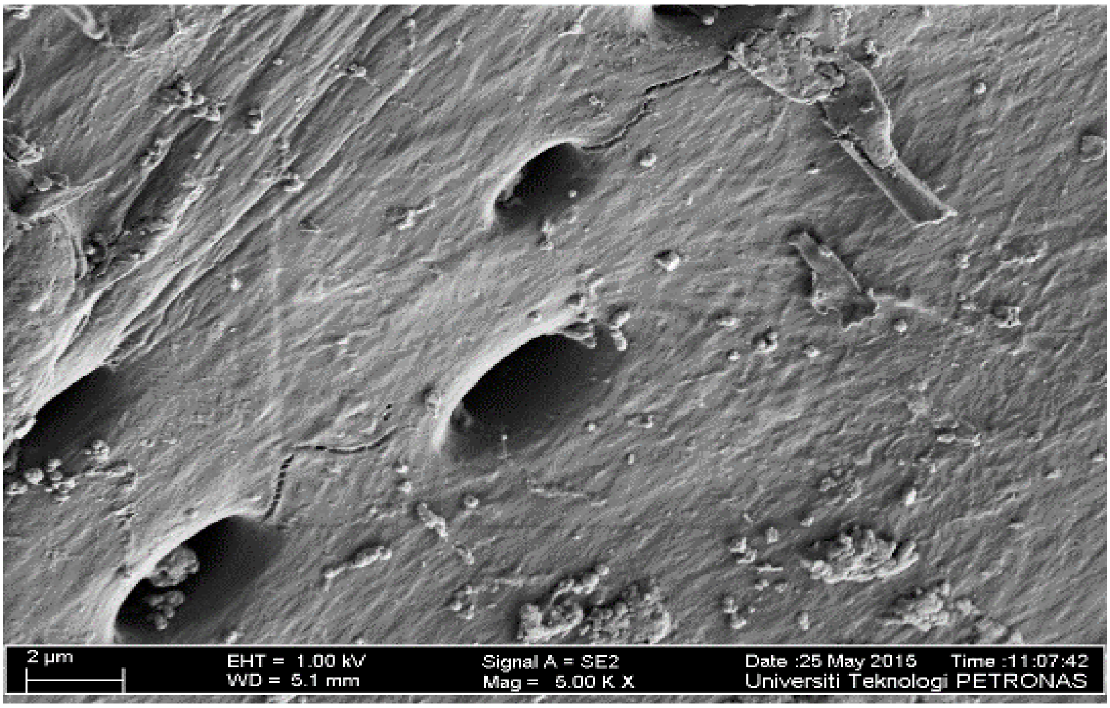


FIGURE 27. RSS raw sample

4.2 Surface Area and Porosity Analysis

TABLE 12. Preparation Condition and Result of Rubber Seed Shell Activated Carbon

Sample	**Act. Time (min)**	**Act. Temp (˚C)**	**Impregnation ratio**	**Specific surface area S_{BET}, (m^2g^{-1})**	**Total pore volume, $V_T(m^3g^{-1})$**	**Average pore diameter, D (nm)**
raw	raw	raw	-	0.67	0.002	5.80
A1	180	500	1:2	24.65	0.041	13.08
B1	180	600	1:2	28.17	0.081	17.30
C1	150	500	1:2	20.93	0.025	14.26
D1	150	600	1:2	23.48	0.039	13.98
E1	210	500	1:2	15.59	0.021	6.48
F1	210	600	1:2	18.92	0.032	8.52
G1	120	500	1:2	6.74	0.008	5.18
H1	120	600	1:2	12.27	0.012	5.94
I1	90	500	1:2	2.08	0.003	4.67

Based on the Table 12 above shows the collected result for the surface area and pore size of RSS activated carbon. Based on the result collected, sample B1 showed the highest surface area and pore volume followed by sample A1 and C1. The formation of surface area and pore volume achieved at optimum temperature of 600˚C for 180 minutes. To produce an activated carbon, the optimum activation temperature are between 400 – 500˚C [47]. Further activation up to 210 minutes also can cause the surface erosion like sample E1 and F1. Sample I1 showed the poorest production of activated carbon. It is because the time allocated during carbonization is not long enough for the activated carbon to form a good surface area and pore diameter. At 500˚C of activation temperature will develops the elimination of molecular weight volatile compounds and further increase the new pore formation while increasing the activation temperature to 600˚C will give an extreme heat to the carbon causing in the knocking and breaking of some porous wall and blocking the formation of porosity [46]. By comparing the result, only activated carbon sample of A1, B1, C1 and raw will be chosen for further analysis to study in the performance of activated carbon to remove the heavy metals.

4.3 Nitrogen Adsorption-Desorption Isotherm

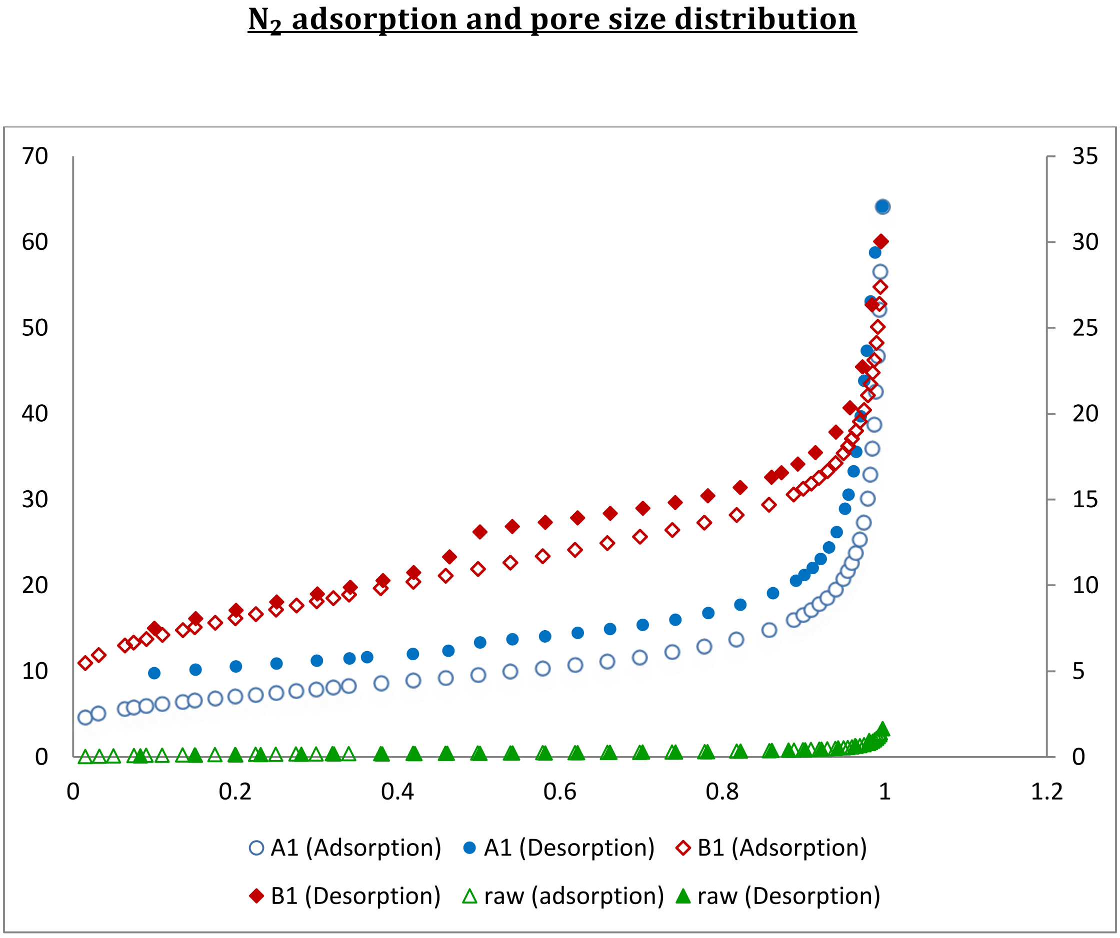


FIGURE 28. Nitrogen Adsorption-Desorption Isotherm

Figure 28 above shows the nitrogen adsorption-desorption analysis for sample A1, B1 and D1. Based on IUPAC, the objective of this study is to categorize the right adsorption isotherm type based on IUPAC classification as shown in Figure 29 below.

To satisfy the analysis of adsorption, the isotherm of adsorption must be fit at least one or a combination of six distinct isotherm graphs [1].

From Figure 28 above, sample B1 are identified similar with TYPE II isotherm. While sample A1 shown similarity with TYPE III. TYPE II isotherm means that the development of unrestricted layer after the monolayer complete was detected in adsorbents with various dimensions of pores and basically this type of isotherm refers to poly-molecular adsorption in either nonporous or macro-porous adsorbents and it do not exhibit the limit of saturation [1]. TYPE III displays features of nonporous sorbents with small energy of adsorbent-adsorbate contact. From figure above, the adsorption isotherm with the highest surface area yield is sample B1.

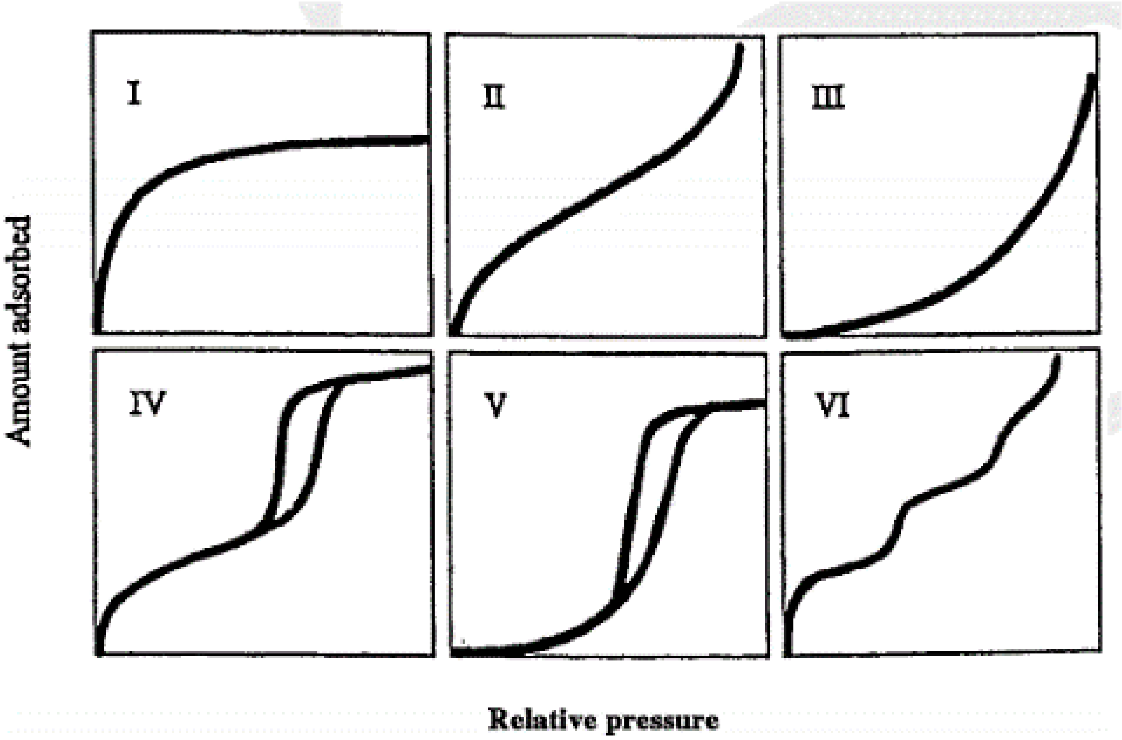


FIGURE 29. IUPAC Classification

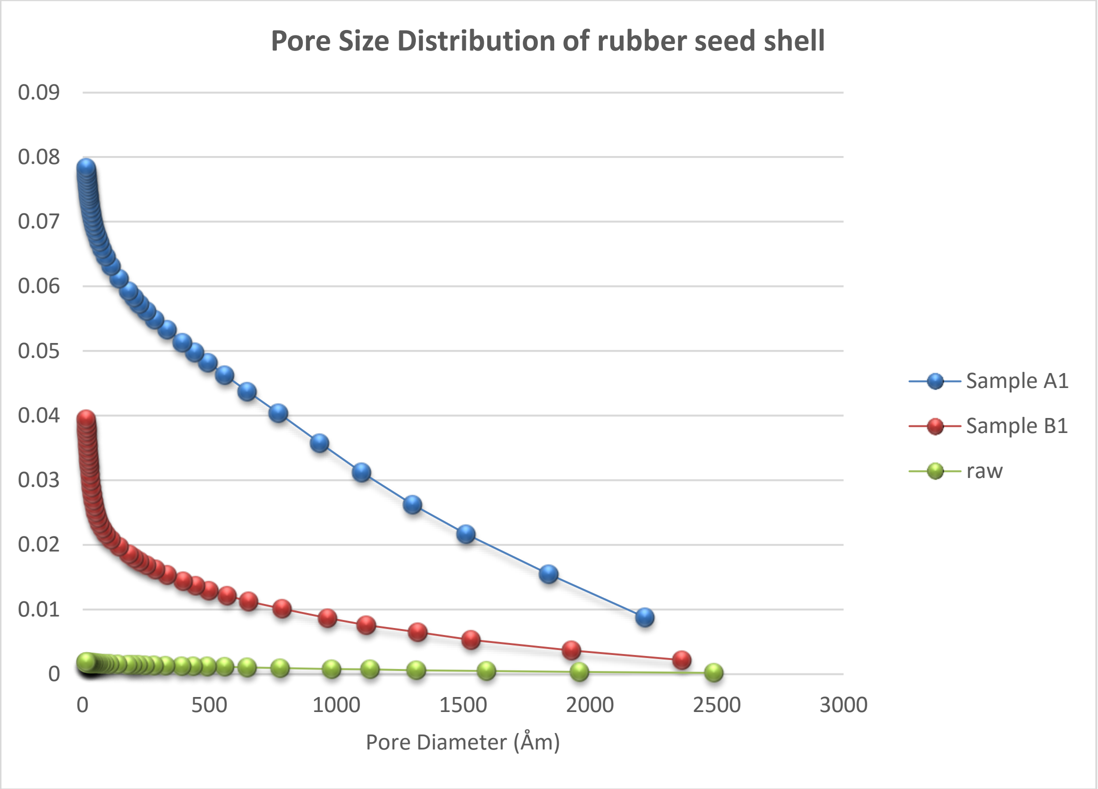


FIGURE 20. Pore Size Distribution of Rubber Seed Shell

The selected samples for the pore size distribution are showed in the Figure 30 for sample A1, sample B1 and raw RSS. Increasing in temperature will increase the formation of mesopore [46]. When the temperature increases, the micropores were enlarged and the wall between the pores collapsed and formed mesopores by classifications accordance to IUPAC that is micropores (diameter (d)<20Å), mesopores (20Å<d<500Å) and macropores (d>500Å) [46]. For sample B1, the average pore diameter determined by BJS method was 173Å if pore diameter falls between ranges of mesopores.

4.4 Removal of Heavy Metal

To test the performance of the activated carbon on heavy metal removal, the solution selected are zinc (II) sulphate and copper (II) sulphate. The sample used only the 500µm because it gave the highest adsorption rate than 1mm samples. Based on Table 4.1, the experiment conducted using the best sample of activated carbon which are sample A1 and sample B1 while the raw rubber seed shell (RSS) also used in this experiment to test their performance in heavy metal removal. Thus, the removal of heavy metals were conducted based on three (3) effects of heavy metals removal which are:

i. Effect of initial concentration
ii. Effect of stirring rate
iii. Effect of contact time

4.4.1 Effect of Initial Concentration

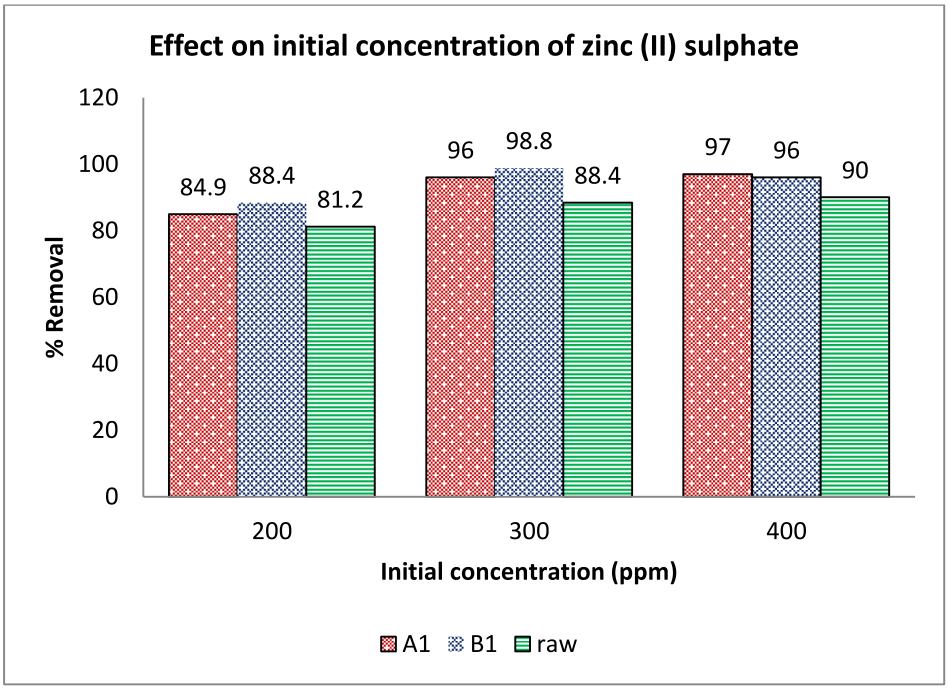


FIGURE 31. Effect on Initial Concentration on Zinc (II) Sulphate
(Mass = 0.05g, Time = 30 min, temperature = 30˚C, stirring rate = 200 rpm)

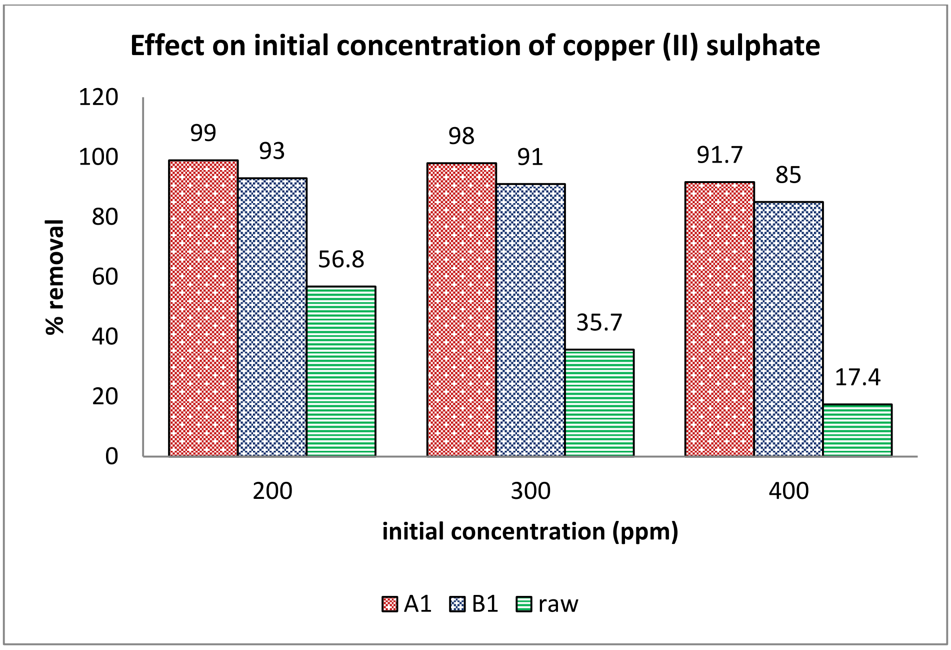


FIGURE 32. Effect on Initial Concentration on Copper (II) Sulphate
(Mass = 0.05g, Time = 30 min, temperature = 30˚C, stirring rate = 200 rpm)

Figure 31 shows the performance of sample A1, B1 and raw RSS in removing zinc in the solution of zinc (II) sulphate. Sample B1 shows the highest removal at 300ppm about 98.8% but decrease about 2.8% to 96% at 400ppm of heavy metal concentration. While sample A1 shows increasing order of percentage removal. Starting with 84.9% of removal on 200ppm of concentration, increased to 96% of removal at 300ppm and the percentage of removal increased up to 97% at 400ppm. The raw sample of RSS also can remove zinc up to 90% at 400ppm. As increasing the heavy metal concentration, the percentage of removal heavy metal should decreased. This is caused by human error while conducting the experiment.

Figure 32 shows the removal of copper. Sample A1 shows the highest percentage of removal on 200ppm of concentration which are 99% and the percentage removal decrease as concentration of heavy metal solution increase. Sample B1 shows about 93% of zinc removal on 200ppm of heavy metal concentration and it keep decreasing as increasing the concentration of the solution. The raw RSS also can remove the zinc about 56.8% on 200ppm concentration.

For initial concentration, sample B1 can removed zinc up to 98.8% which is better than other sample while for copper, sample A1 showed the highest removal up to 99%. Based on Table 4.1, sample B1 showed the highest surface area $28.71 m^2 g^{-1}$, sample A1 with $24.65 m^2 g^{-1}$ and raw sample is $0.67 m^2 g^{-1}$. Since zinc have bigger size than copper, thus, for effect of initial concentration, sample B1 can removed zinc ion better and sample A1 more suitable to remove copper ions than other samples. The higher the concentration of the heavy metal solution, the lower the percentage of heavy metal removal. This is because of the heavy metal ion no longer attached to the surface of the adsorbent since the adsorbent have reached their limited surface to adsorb the metal ions.

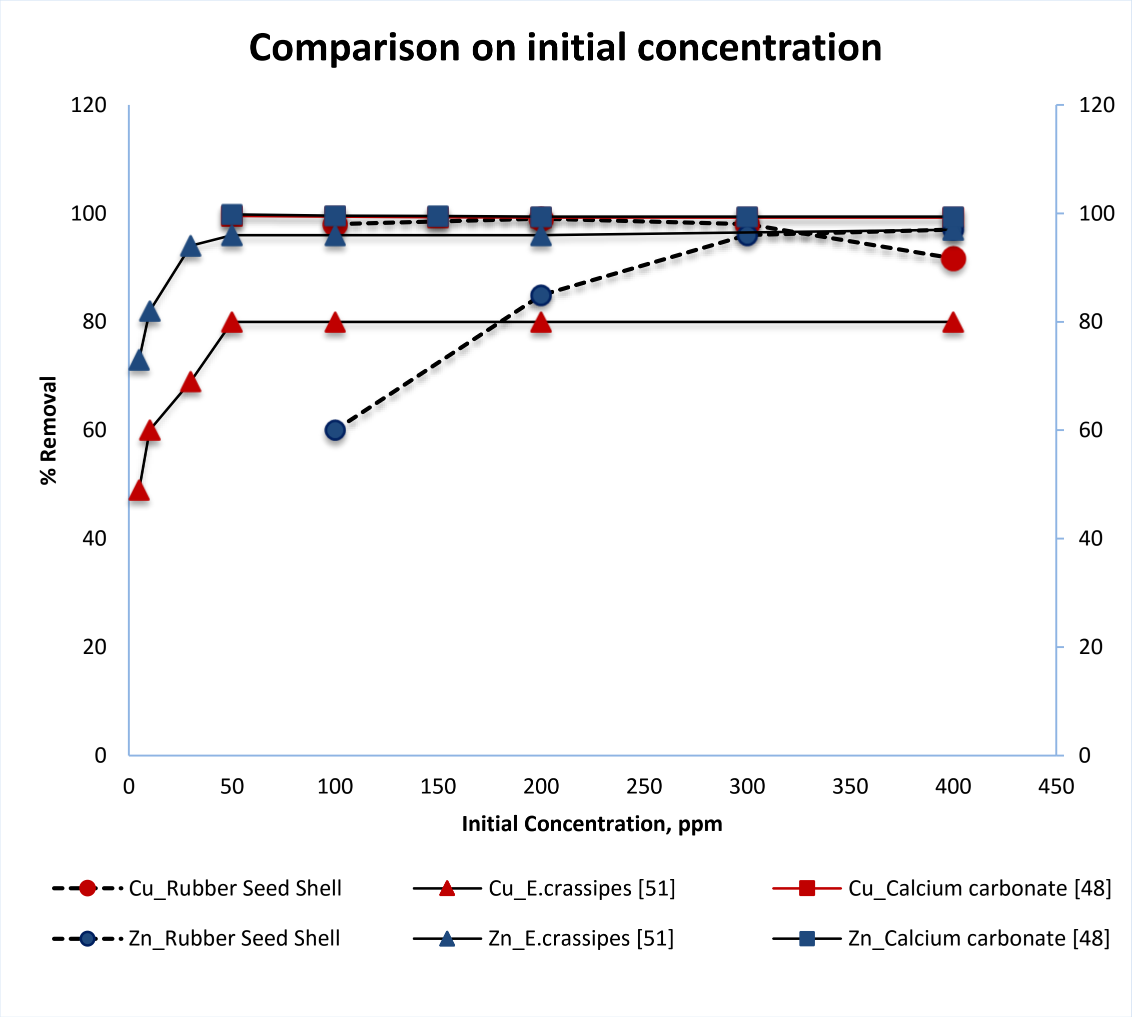


FIGURE 33. The Comparison Result on Initial Concentration Effect with Other Adsorbent

For initial concentration effect, sample B1 was chosen to compare with other researches using other adsorbents like *Eichhornia crassipes (E.crassipes)* or water hyacinth and calcium carbonate. From Figure 4.10, for the comparison on zinc removal, rubber seed shell (RSS) falls the second highest removal after calcium carbonate while *E.crassipes* showed the lowest removal of zinc. Calcium carbonate is an adsorbent from laboratory scale used by Khalil Ahmad [48] to remove the heavy metals from aqueous solution. The removal of zinc by using calcium carbonate is about 99.8% at 45ppm concentration of aqueous solution and slightly decrease to 99.5% of removal at 145ppm concentration. Using RSS sample B1 with highest surface area, the removal of zinc is up to 98.8% at 300ppm concentration and decreased to 96% at 400ppm concentration of zinc (II) sulphate solution. While, using adsorbent derived from *E.crassipes,* the removal of zinc is up to 96% at 50ppm of heavy metal solution.

For copper removal comparison, activated carbon derived from RSS also shows the second highest percentage of removal after calcium carbonate. The difference methods on experiment conducted contributed with different outcomes of removal. Using calcium carbonate, copper can be removed up to 99.5% at 55ppm concentration of heavy metal solution and slightly decreased to 99.2% at 190ppm concentration of heavy metal solution. Using sample A1 of RSS, copper can be removed up to 99% on 200ppm concentration and decreased to 91.7% at 400ppm concentration of copper (II) sulphate solution. While, using *E.crassipes,*it removed copper up to 80% at 50ppm concentration of heavy metal solution. At lower concentration, adsorption is higher due to high collision efficiency between heavy metal ions and the adsorbent [48]. But, for economic point, RSS have a good selling point because RSS easily to find since rubber plantation is the second largest plantation in Malaysia rather than *E.crassipes* which only can get at aquatic place while calcium carbonate only can get from the laboratory and quite expensive than RSS.

4.4.2 Effect of Stirring Rate

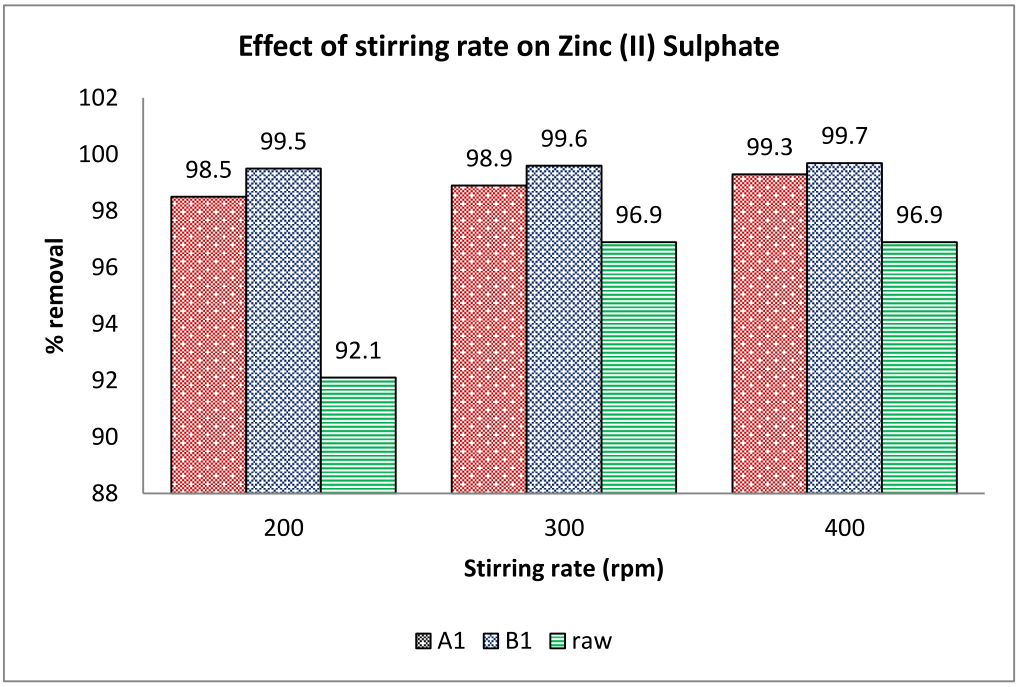


FIGURE 34. Effect of Stirring Rate on Zinc (II) Sulphate
(Concentration = 100ppm, mass = 0.5g, time = 1h, temperature = 30˚C)

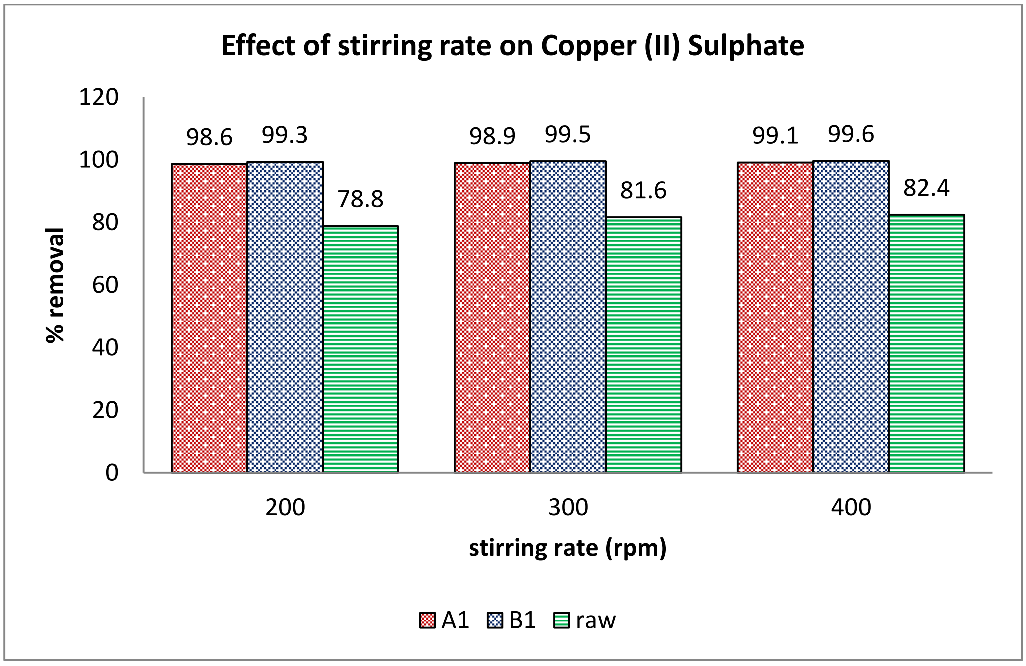


FIGURE 35. Effect of Stirring Rate on Copper (II) Sulphate
(Concentration = 100 ppm, mass = 0.5g, time = 1h, temperature = 30˚C)

Figure 34 shows the removal of zinc from zinc (II) sulphate solution. From the graph, sample B1 shows the highest percentage of removal followed with sample A1 and raw RSS sample. For sample B1, the percentage increasing from 99.5% at 200rpm to 99.7% at 400rpm of stirring rate. For sample A1, the percentage removal also increased as increasing stirring rate which is 98.5% at 200rpm to 99.3% at 400rpm. The pattern of percentage removal same on raw sample of RSS where because of some pore volume exist on the surface, the percentage increased from 92.1% at 200rpm to 96.9% at 400rpm of stirring rate.

Figure 35 shows the removal of heavy metal copper from copper (II) sulphate. The percent of copper removal shows same result like zinc removal where sample B1 shows highest percentage of removal followed with sample A1 and raw RSS sample. For sample B1, the percentage removal is 99.3% at 200rpm increased to 99.6% at 400rpm of stirring rate. Sample A1 shows the percentage of copper removal about 98.6% at 200rpm increased to 99.1% at 400rpm of stirring rate. While for raw sample, the percentage at 200rpm is 78.8% increased to 82.4% at 400rpm of stirring rate.

With highest surface area and pore diameter, sample B1 can removed both zinc and copper better under effect of stirring rate. Due to the agitation, it developed a proper contact between heavy metal solutions with the activated carbon [5]. The increasing of stirring rate will increase the percentage of heavy metal removal which is the higher the stirring rate it will improve the diffusion of metal ions to the activated carbon surface and also it initiated drop in the film boundary layer around the activated carbon [49].

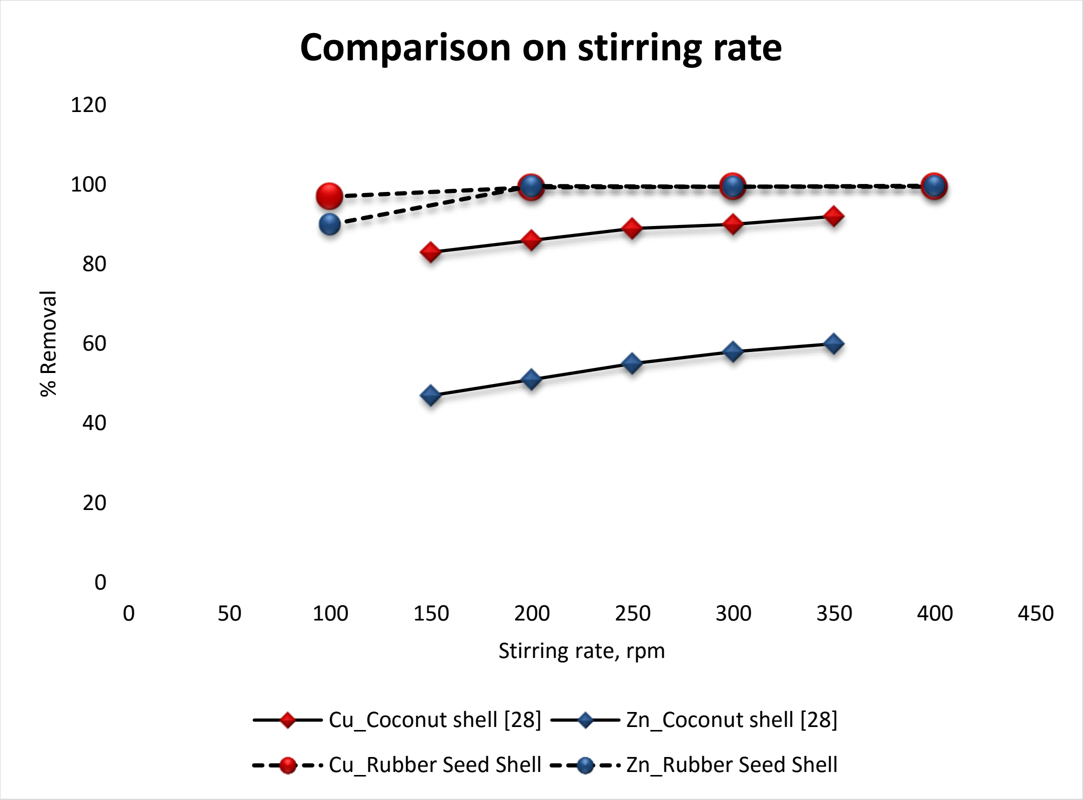


FIGURE 36. The Comparison Result on Stirring Rate Effect with Other Adsorbent

Sample B1 was chosen to be compared with other adsorbent which is activated carbon derived from coconut shell. Coconut shell also one of low-cost agriculture waste used to be a good adsorbent. For stirring rate effect, sample B1 shows the highest percentage for both zinc and copper removal. For zinc removal, the percentage of removal is 99.7% at 400rpm and for copper removal, the percentage of removal is up to 99.6% at 400rpm of stirring rate. While, using adsorbent derived from coconut shell, the percentage of zinc removal is up to 62% at 350rpm and the percentage of copper removal is up to 91% at 350rpm of stirring rate. Both RSS and coconut shell removed zinc better than copper. This is because the molecular structure of copper is stable than zinc thus, it is tough to break the bond of the copper ions [50]. Based on their research, 350 rpm is the maximum recoveries of the heavy metal removal where increasing the stirring rate improved the metals ions dispersion to the external of the adsorbent and also triggered decline in the film boundary cover around the adsorbent [28].

4.4.3 Effect of Contact Time

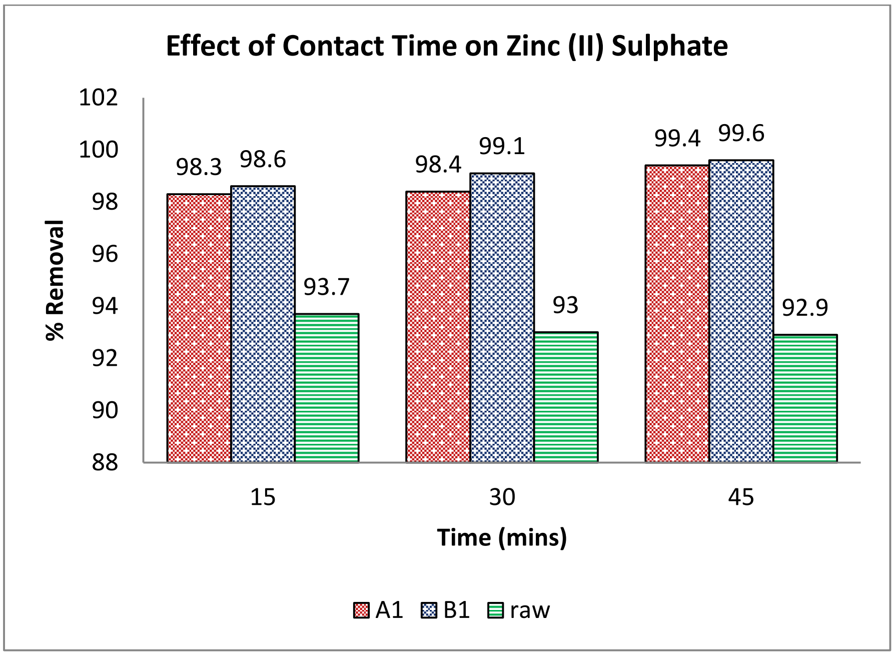


FIGURE 37. Effect of Contact Time on Zinc (II) Sulphate

(Concentration = 100ppm, mass = 0.1g, temperature = 30˚C, stirring rate = 300 rpm)

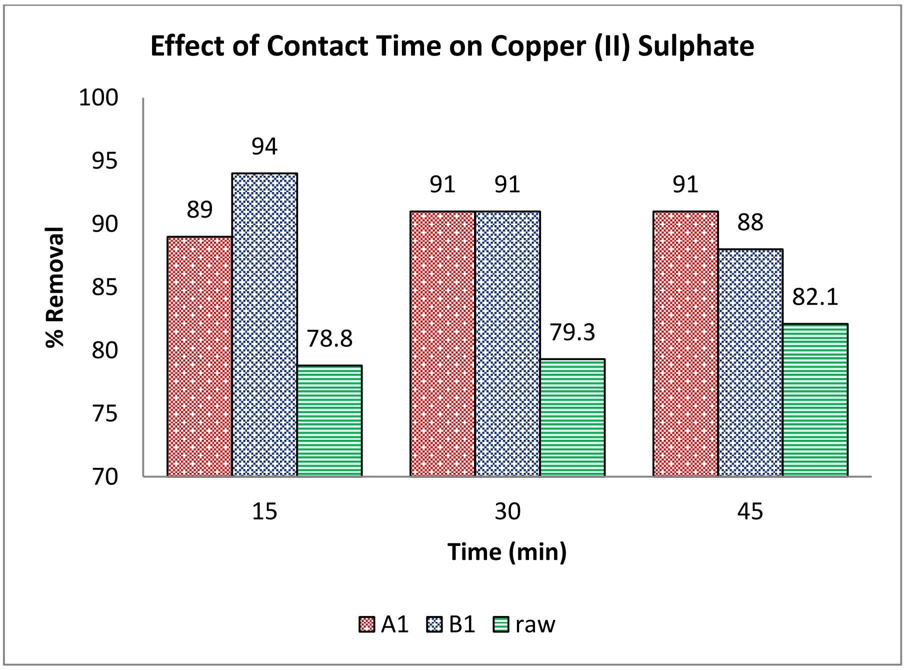


FIGURE 38. Effect of Contact Time on Copper (II) Sulphate

(Concentration = 100ppm, mass = 0.1g, temperature = 30˚C, stirring rate = 300 rpm)

From Figure 37 is the effect of contact time on zinc removal from zinc (II) sulphate solution. From the graph, sample B1 shows the highest percentage of zinc removal followed with sample A1 and raw RSS. For sample B1, at minutes of 15, the percentage of zinc removal is 98.6% and increased up to 99.6% at 45 minutes of contact time. Sample A1 shows 98.3% of zinc removal at minutes of 15 and the percentage increased to 99.4% at minutes of 45. While raw sample of RSS shows decreased order of percentage removal which is at minutes of 15, the percent of zinc removal is 93.7% and it decreased to 92.9% at minutes of 45.

Figure 38 is the graph of copper removal from copper (II) sulphate solution. Sample B1 shows the highest trending of copper removal followed with sample A1 and raw RSS. At minutes of 15, sample B1 removed copper up to 94% but decreased to 88% at minutes of 45. Sample A1 removed 89% of copper at minutes of 15 and it increased to 91% on minutes of 30 and 45 of contact time. It shows a constant percentage of removal where the adsorbent already achieved the optimum time to adsorb more heavy metal ions. After some time, may be it is hard to conquer the residual spots of the activated carbon because of the adsorbate ions on the solid and in the heavy metal solution [5]. Thus, increasing in contact time to promote removal of the heavy metal did not bring any changes in the percentage but it resulted in desorption of the metal ions from the surface of the activated carbon [28]. This effect conducted because different heavy metal ions attained equilibrium at different times [28]. While for raw sample, the percentage of copper removal is 78.8% at minutes of 15 and increased to 82.1% at minutes of 45.

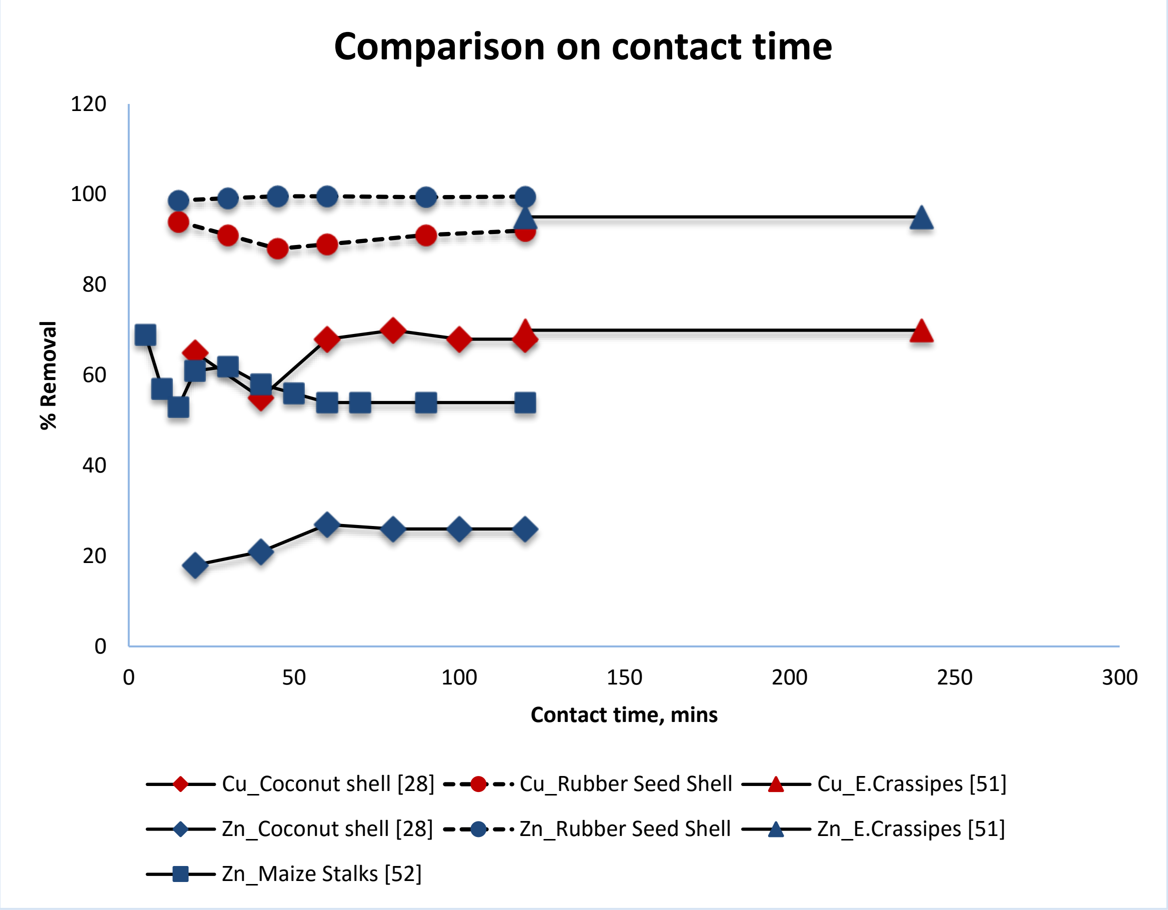


FIGURE 39. Comparison of Contact Time Effect with Other Adsorbent

For effect of contact time, sample B1 was chosen to be compared with other adsorbents which are coconut shell, *E.crassipes*, and maize stalk. For zinc removal, sample B1 shows the highest percentage of removal followed with *E.crassipes*, maize stalks and coconut shell. All the adsorbents already achieved the optimum time thus, increasing the time will not increase the percentage of removal but resulted in desorption of the metal ions from the surface of adsorbents. After achieved optimum time, sample B1 of RSS can removed zinc up to 99.5% up to minutes of 100. For adsorbent derived from *E.crassipes*, the percent of zinc removal is 94% at minutes of 240. Maize stalk adsorbent records the percentage of removal up to 54% at minutes 120. While coconut shell adsorbent, the percentage of zinc removal is 24% at minutes of 120.

For removal of copper, sample B1 also shows the highest percentage removal followed with *E.crassipes* and coconut shell. For sample B1, after minutes of 100, the percentage of copper removal is recorded at 92%. For adsorbent derived from *E.crassipes,* the percentage of copper removal is achieved 68% at minutes of 240. While adsorbent derived from coconut shell also removed copper up to 68% at minutes of 120. Based on comparison of all adsorbent, RSS is the raw sample that easily to find in Malaysia. Besides it only need low cost to prepare the activated carbon since it only used sodium hydroxide as activating agent.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The agricultural abundance wastes of rubber seed shell have a high adsorption capacity when it is activated by Sodium Hydroxide (NaOH) solution. The optimum condition for the heavy metal ions removal is at contact time 30 min and adsorbent dosage of 1000mg/100mL. The BET surface are of activated carbon, the total pore volume and average pore diameter will be analysed by the FESEM to find the good condition of activated carbon. The activating temperature will be at 500˚C and 600˚C in range of activation time of 90, 120,150, 180 and 210 minutes.

From the result collected from the removal of zinc and copper, we can see that the best sample achieved almost 100% removal is sample B1. Sample B1 was activated at 600˚C for 180 minutes using 1:2 of raw rubber seed shell and sodium hydroxide solution. The specific surface area of Sample B1 is 28.17 $(\mathrm{m^2g^{-1}})$ with pore volume of 0.0081$(\mathrm{m^3g^{-1}})$. Removal of the heavy metal of zinc is better than copper using the sample B1 up to 99% which is better than other low-cost agriculture waste used by other researches for example banana peel, coconut shell, maize stalks, palm kernel, nutshells and many more.

5.2 Recommendation

The study on suitability of chemical reagent must be suggested in order to vary the properties of different chemical reagents. Different chemical reagent will result in different rate of heavy metal adsorption. In future, an investigation on different wastes can be done at the same time to compare the adsorption rate, the activation time and temperature, the pH and carbon content in each waste such as palm oil, coconut husk or rice husk.

Besides, for the time given for students to undergo their experiment and researches should be given more time especially for students doing the researches on removal of heavy metal ion, biodegradation and others. If possible, the universities parties should allow the student to start the experiment during their FYP I to make sure they can use the time available to finish their experiment.

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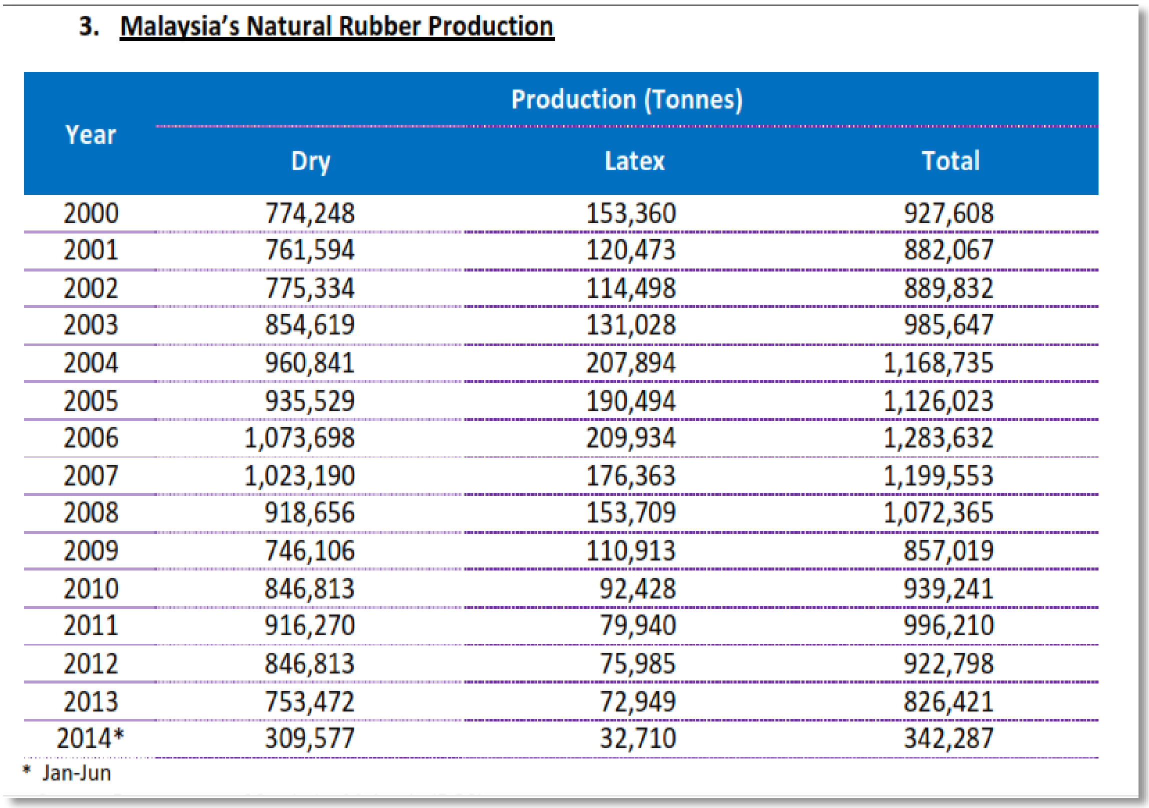
APPENDICES

1. Malaysia's natural rubber production

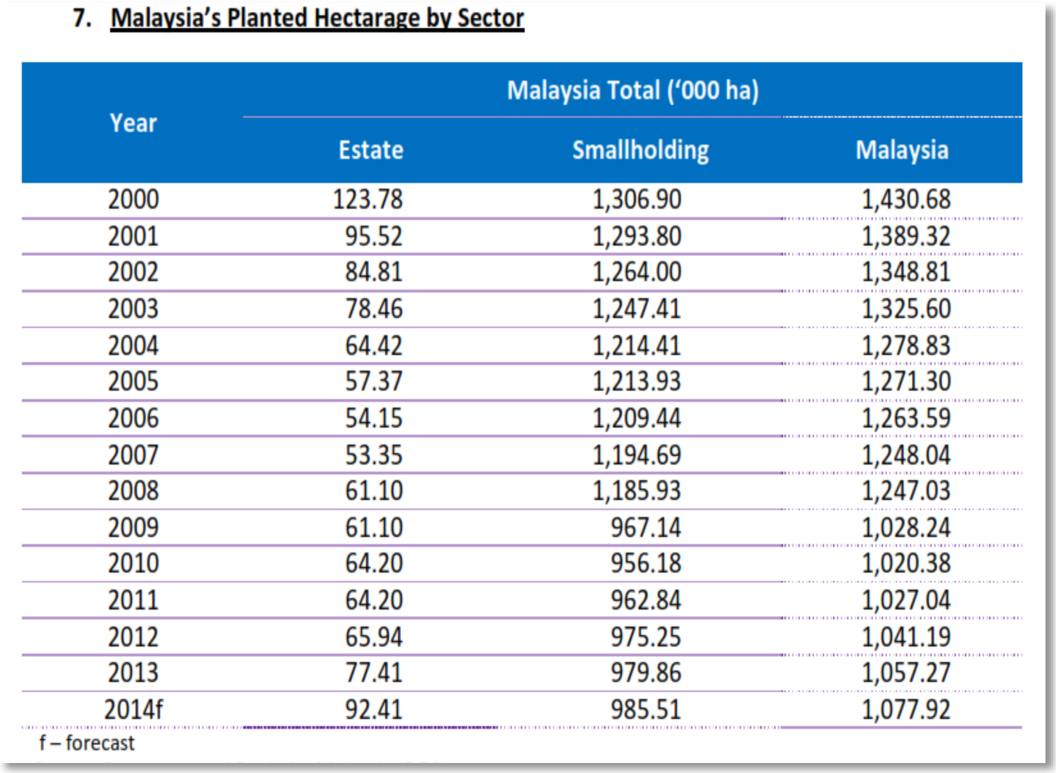


3. Malaysia's Natural Rubber Production

Year	Production (Tonnes)		
	Dry	Latex	Total
2000	774,248	153,360	927,608
2001	761,594	120,473	882,067
2002	775,334	114,498	889,832
2003	854,619	131,028	985,647
2004	960,841	207,894	1,168,735
2005	935,529	190,494	1,126,023
2006	1,073,698	209,934	1,283,632
2007	1,023,190	176,363	1,199,553
2008	918,656	153,709	1,072,365
2009	746,106	110,913	857,019
2010	846,813	92,428	939,241
2011	916,270	79,940	996,210
2012	846,813	75,985	922,798
2013	753,472	72,949	826,421
2014*	309,577	32,710	342,287

* Jan-Jun

2. Malaysia’s Planted Hectarage by sector

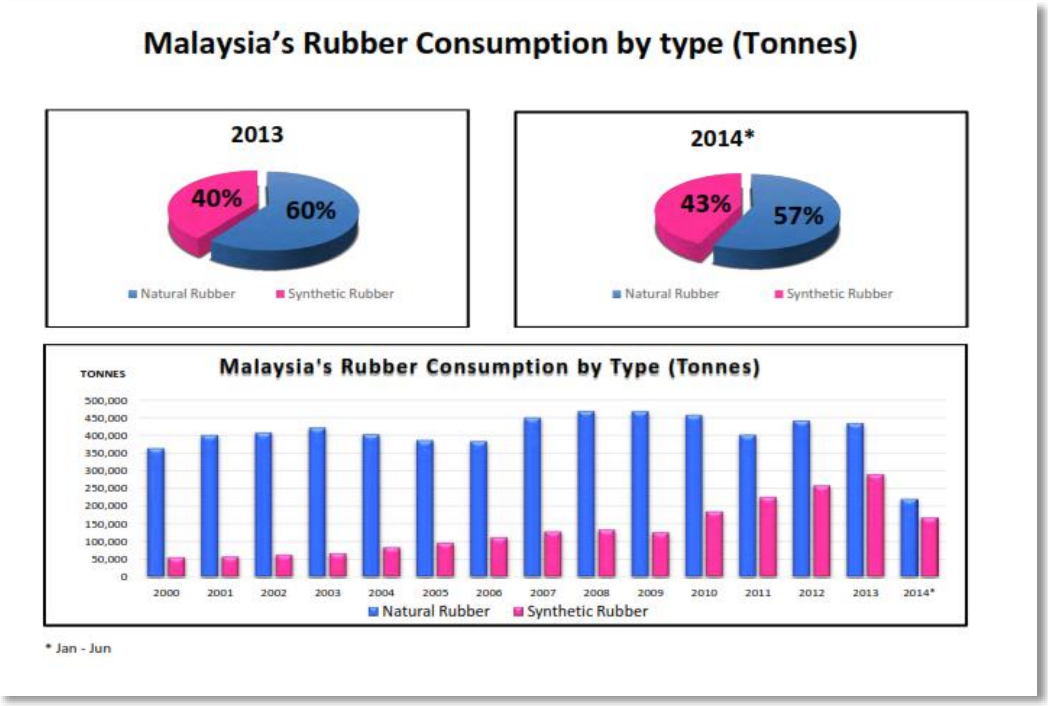


7. Malaysia’s Planted Hectarage by Sector

Year	Malaysia Total (‘000 ha)		
	Estate	Smallholding	Malaysia
2000	123.78	1,306.90	1,430.68
2001	95.52	1,293.80	1,389.32
2002	84.81	1,264.00	1,348.81
2003	78.46	1,247.41	1,325.60
2004	64.42	1,214.41	1,278.83
2005	57.37	1,213.93	1,271.30
2006	54.15	1,209.44	1,263.59
2007	53.35	1,194.69	1,248.04
2008	61.10	1,185.93	1,247.03
2009	61.10	967.14	1,028.24
2010	64.20	956.18	1,020.38
2011	64.20	962.84	1,027.04
2012	65.94	975.25	1,041.19
2013	77.41	979.86	1,057.27
2014f	92.41	985.51	1,077.92

f – forecast

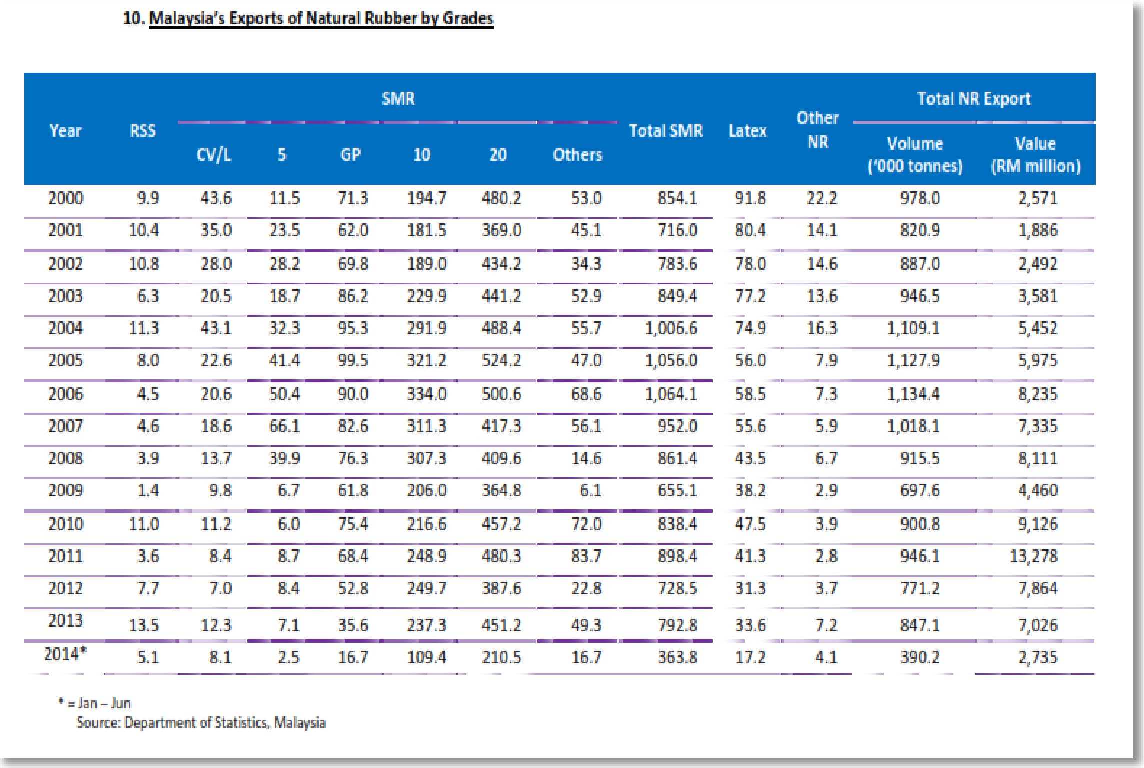
3. Natural rubber consumption by types in Malaysia



Malaysia’s Rubber Consumption by type (Tonnes)

* Jan - Jun

4. Natural rubber export by grades



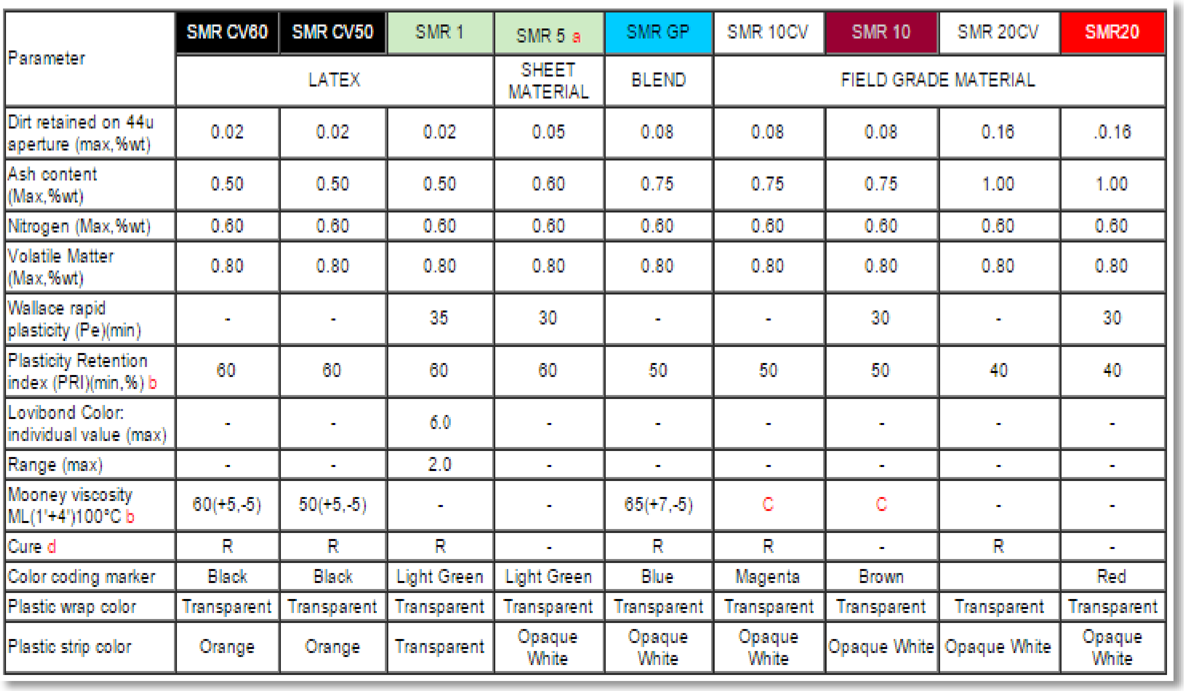
10. Malaysia's Exports of Natural Rubber by Grades

Year	RSS	SMR						Total SMR	Latex	Other NR	Total NR Export	
		CV/L	5	GP	10	20	Others				Volume ('000 tonnes)	Value (RM million)
2000	9.9	43.6	11.5	71.3	194.7	480.2	53.0	854.1	91.8	22.2	978.0	2,571
2001	10.4	35.0	23.5	62.0	181.5	369.0	45.1	716.0	80.4	14.1	820.9	1,886
2002	10.8	28.0	28.2	69.8	189.0	434.2	34.3	783.6	78.0	14.6	887.0	2,492
2003	6.3	20.5	18.7	86.2	229.9	441.2	52.9	849.4	77.2	13.6	946.5	3,581
2004	11.3	43.1	32.3	95.3	291.9	488.4	55.7	1,006.6	74.9	16.3	1,109.1	5,452
2005	8.0	22.6	41.4	99.5	321.2	524.2	47.0	1,056.0	56.0	7.9	1,127.9	5,975
2006	4.5	20.6	50.4	90.0	334.0	500.6	68.6	1,064.1	58.5	7.3	1,134.4	8,235
2007	4.6	18.6	66.1	82.6	311.3	417.3	56.1	952.0	55.6	5.9	1,018.1	7,335
2008	3.9	13.7	39.9	76.3	307.3	409.6	14.6	861.4	43.5	6.7	915.5	8,111
2009	1.4	9.8	6.7	61.8	206.0	364.8	6.1	655.1	38.2	2.9	697.6	4,460
2010	11.0	11.2	6.0	75.4	216.6	457.2	72.0	838.4	47.5	3.9	900.8	9,126
2011	3.6	8.4	8.7	68.4	248.9	480.3	83.7	898.4	41.3	2.8	946.1	13,278
2012	7.7	7.0	8.4	52.8	249.7	387.6	22.8	728.5	31.3	3.7	771.2	7,864
2013	13.5	12.3	7.1	35.6	237.3	451.2	49.3	792.8	33.6	7.2	847.1	7,026
2014*	5.1	8.1	2.5	16.7	109.4	210.5	16.7	363.8	17.2	4.1	390.2	2,735

* = Jan – Jun

Source: Department of Statistics, Malaysia

5. Natural rubber grades in Malaysia



Parameter	SMR CV60	SMR CV50	SMR 1	SMR 5 a	SMR GP	SMR 10CV	SMR 10	SMR 20CV	SMR20
	LATEX			SHEET MATERIAL	BLEND	FIELD GRADE MATERIAL			
Dirt retained on 44u aperture (max,%wt)	0.02	0.02	0.02	0.05	0.08	0.08	0.08	0.16	.0.16
Ash content (Max,%wt)	0.50	0.50	0.50	0.60	0.75	0.75	0.75	1.00	1.00
Nitrogen (Max,%wt)	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60
Volatile Matter (Max,%wt)	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80
Wallace rapid plasticity (Pe)(min)	-	-	35	30	-	-	30	-	30
Plasticity Retention index (PRI)(min,%) b	60	60	60	60	50	50	50	40	40
Lovibond Color: individual value (max)	-	-	6.0	-	-	-	-	-	-
Range (max)	-	-	2.0	-	-	-	-	-	-
Mooney viscosity ML(1'+4')100°C b	60(+5,-5)	50(+5,-5)	-	-	65(+7,-5)	C	C	-	-
Cure d	R	R	R	-	R	R	-	R	-
Color coding marker	Black	Black	Light Green	Light Green	Blue	Magenta	Brown		Red
Plastic wrap color	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
Plastic strip color	Orange	Orange	Transparent	Opaque White	Opaque White	Opaque White	Opaque White	Opaque White	Opaque White

6. Yield percentage of activated carbon

Sample A1:

$raw = 18.74g$

$NaOH = 38.7332g$

Before carbonization:

$weight\ of\ boat = 95.21g$

$weight\ of\ boat + sample = 167.92g$

After carbonization

$weight\ of\ sample = 15.58g$

After washed sample with hot distilled water

$weight\ of\ sample = 7.5296g$

$$yield\ percentage\ of\ sample\ B1 = \frac{15.58 - 7.5296}{15.58} = \mathbf{51.67}\%$$

Sample B1:

$raw = 25.0394g$

$NaOH = 50.1572g$

Before carbonization:

$weight\ of\ boat = 95.30g$

$weight\ of\ boat + sample = 157.96g$

After carbonization

$weight\ of\ sample = 12.4272g$

After washed sample with hot distilled water

$weight\ of\ sample = 6.5238g$

$$yield\ percentage\ of\ sample\ B1 = \frac{12.4272 - 6.5238}{12.4272} = \mathbf{47.5}\%$$

Sample C1:

$raw = 19.75gg$

$NaOH = 40.4555g$

Before carbonization:

$weight\ of\ boat = 95.23g$

$weight\ of\ boat + sample = 160.54g$

After carbonization

$weight\ of\ sample = 14.03g$

After washed sample with hot distilled water

$weight\ of\ sample = 6.7193g$

$$yield\ percentage\ of\ sample\ B1 = \frac{14.03 - 6.7193}{14.03} = \mathbf{47.89}\%$$