ADSORPTION OF HEAVY METAL USING SLUDGE ACTIVATED CARBON DERIVED FROM SLUDGE

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Civil Engineering

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Civil Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CIVIL ENGINEERING)

Approved by,

____

(AP Dr. Shamsul Rahman Mohamed Kutty)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
SEPT 2017

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.

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ABSTRACT

The amount of sludge produced by the domestic and sewage company is about 5.3 million^3 per year and most of the sludge are being wasted in an unused landfill. This study proposed dry activated sludge as low-cost adsorbents for the Zn(II) from the wastewater. It is focused on the adsorption of Zn(II) by sludge biomass-based activated carbon which will be conducting using batch tests. The effect of contact time, initial metal ions concentration and adsorbent dosage on the removal of Zn(II) from the synthetically produced wastewater were investigated. The removal efficiency was found to have relations with the initial Zn (II) concentration, contact time as well as adsorbent dosages of the adsorbents. The adsorption kinetics of the heavy metal ion, Zn(II) by SAC adsorbent were found to follow pseudo-second order kinetic models indicates that the experiment undergone chemisorption process. The Freundlich isotherm described Zn(II) adsorption by SAC were slightly better than Langmuir isotherm which yielded high 2 values from 0.9905 to 1.000. The best sample achieved beyond 50% removal is sample with 10.0g of adsorbent dosage in 100mg/L concentration of zinc metal solution. SAC is made up of small heterogeneous adsorption patches which are similar to each other with respect to adsorption phenomenon. [16,18]. Besides that, the *n* values obtained from the Freundlich isotherm plot which describing the factor that related to capacity and intensity factor showing they are less than 1 and this indicates that the heavy metals adsorption are favourable. From the values, it shows that the heavy metals uptake by SAC are higher and applicable in removing of the heavy metal.

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ABBREVIATION AND NOMENCLATURE

UTP	: Universiti Teknologi Petronas
STP	: Sewage Treatment Plant
AS	: Activated Sludge
SAC	: Sludge Activated Carbon
DAS	: Dry Activated Sludge
TSS	: Total Suspended Solid
TSP	: Total Suspended Particulates
BOD	: Biological Oxygen Demand
COD	: Chemical Oxygen Demand
DOE	: Department of Environmental

Chapter 1

Introduction

1.1 Background of Study

The amount of sludge produced by the sewage treatment plant increases annually and this will cause a secondary pollution in the environment without proper treatment and disposal. Many conventional disposal methods have been applied such as landfill, waste incineration and as well as sea dumping. However, past researchers found out that these methods are less cost effective and innovative solution in order to remove sludge disposal.

As for this project, sludge that are produced by the UTP Sewage Treatment Plant (STP) is passed through the digestion chamber where anaerobic decomposition occur which turned into liquefaction form thus making the volume of the sludge reduced. After digested sludge is produced, it is placed in a drying bed to be delivered to the Indah Water Konsortium (IWK) landfill which is located in Kuala Lumpur.

However, people put less interest in increasing of energy cost, ash disposal and gaseous emission which are widely used nowadays. A better solution that has cost effective must be done in order to solve the sludge disposal issue.

FIGURE 1: Landfill spaces located in Kuala Lumpur

Many researchers have tried different approach in converting dry activated sludge into dry activated carbon which are better removal of heavy metal from aqueous solution. This is because increases in water contamination give strong attention towards people. The research for the environmental problem has been focused mainly on municipal and industrial wastewater due potential impact on human. Most of the trace metals found are non-biodegradable which can occur in both environment and biological system. The metals may lead to various disease such as cancer and damage in the nervous system that can occur in human body (Leone et al., 2006).

Due to this matter, living or non-living adsorbents are tested for the removal of heavy metal by using adsorption process. Physical and chemical interactions can be seen under the surface adsorption of mechanism that occur between metal activated carbon surface and it is clearly effective as assumed in the equilibrium models. (Flouty and Estephane, 2012).

Activated carbon works as a carbonaceous adsorbent with highly developed porous structure and it is also found to be very effective on other methods due to its high quality achievement of the effluent, design integrity, ease of operation and insensitivity to toxic substances. Due to economic crisis nowadays, many investigators have put efforts in lowering the alternative cost to activate the carbon from a range of carbonaceous and mineral precursors, such as fruit stones, nut shells, fly ash, minerals and most importantly from sewage sludge (Pollard et al., 1992) and (Heschel and Klose., 1995).

Many methods have been applied to meet the objectives such as chemical precipitation, coagulation-flocculation, membrane filtration and ion-exchange. However, most of the techniques to remove heavy metals in the wastewater are highly concentrated and less cost-effective that falls under certain cases. Therefore, the adsorption process was chosen to remove the heavy metals due to its utilization of low-cost adsorbent.

The use of adsorbent will contribute to the combination of physical attraction force, chemical binding and exchange of ions that is defined as bio-adsorption process in order to remove metal by the utilization of biological material. It has been proven by the researchers that the biosorption process are extremely effective process to remove heavy metals in wastewater.

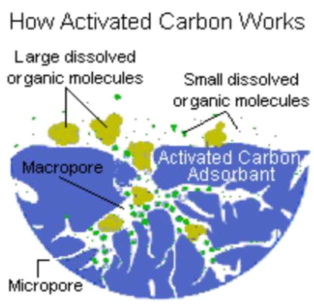

FIGURE 2 : How Activated Carbon Works & Samples of Dry Sewage Sludge

1.2 Problem Statement

Based on the statistics obtained from IWK report, (Kaur, Wani, Singh, Lal, 2011), based on perspective sludge production factor (SPF), the amount of excessive sludge that is wasted by domestic and sewage company on the landfill is about 5.3 $million^3$ per year.

Different conventional methods such as chemical precipitation, ion-exchange, filtration, electro-coagulation and membrane technologies have been used by researchers to successfully remove heavy metals from aqueous solution. These methods however are less effective for the cases where the concentration of metal in solution lies in the range of 1-100mg/L [16]. Furthermore, large amounts of toxic chemical sludge are produced by conventional treatment technologies which increases cost and is not eco-friendly.

The purpose of this research is to produce an alternative solution which is able to optimize the usage of activated sludge from being wasted and polluted by converting the dry activated sludge to Activated Carbon (AC), which is used as an adsorption mechanism to remove Zn(II) in aqueous solution.

1.3 Objective

Several objectives that have been set up for this research are as follows:

1) To produce activated carbon from domestic wastewater treatment sludge.
2) To use the prepared Sludge Activated Carbon (SAC) to treat wastewater that acted with zinc metal ion only.
3) To analyse the adsorption process using Freundlich and Langmuir Isotherm model and pseudo-first order and pseudo-second order kinetic studies.

1.4 Scope of Study

Heavy metals such as zinc is a major pollutant in a wastewater. This study focuses on carbon activation from the dry activated sludge and using Sludge Activated Carbon (SAC) to adsorb Zn(II). To study the effect of initial zinc concentration and contact time, adsorption experiments will be carried out. The process will adopt batch experimental technique and is included with Activated Carbon ash produced from the dry activated sludge waste as adsorbent for zinc removal. By using Spectrophotometer and Zinc Powder Pillow, the concentration of zinc removal is measured. In addition, two kinetic models, pseudo-first-order and pseudo-second-order kinetics adoption will be done to study the mechanism that controls the removal process and the removal capacities of the adsorbent. This is done according to the Langmuir and Freundlich isotherm models.

Precipitation of ion as a metal hydroxide is the most popular technique used to remove soluble metal ions from solution. The pH value of a solution is raised by using materials such as lime, or sodium hydroxide which are common alkaline materials that corresponds metallic hydroxide compounds to become more soluble and precipitate from the solution. Figure below shows the hydroxide solubility curve which explains the solubility of the common heavy metal ions and their respective solubility versus pH.

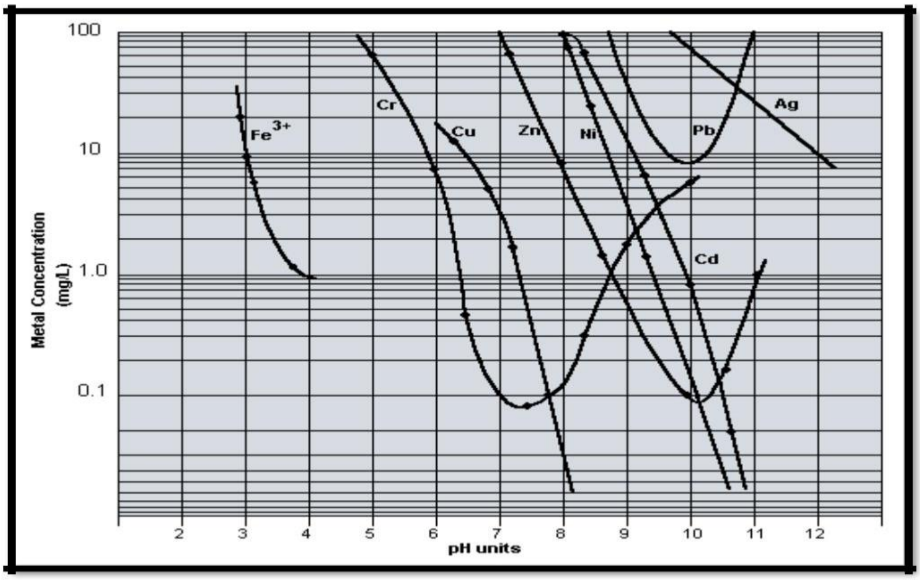

FIGURE 3 : Metal Solubility Curve

1.5 Relevancy and Feasibility Study of the Project

Any study done by a researcher should be beneficial to not only the society but also should bring a change to the world. This topic is relevant as it is studied to:

i) Propose an adsorbent material which is natural and can be used to remove heavy metal;

ii) Find a cheaper alternative to remove heavy metal;

iii) Reducing pollution by discarding toxic and carcinogenic effluent in industrial wastewater.

CHAPTER 2

LITERATURE REVIEW

2.1 Heavy Metals

Heavy metals are generally considered those whose density exceeds 5 g per cubic centimetre. Most of the elements that falls into this category are highly water soluble, well-known toxics and carcinogen agents [3]. Heavy metals are described as any metallic component which has a high density and is poisonous or toxic even at lower concentration [4]. Cadmium (Cd), lead (Pb), cobalt (Co), nickel (Ni), zinc (Zn), iron (Fe), copper (Cu), silver (Ag), arsenic (As) are elements that exist in a heavy metal, along with the elements in the platinum group.

2.2 Sources of heavy metals

Heavy metals natural occurring elements which are found beneath the earth crust. Heavy metals not only contaminate the environment, its exposure is harmful and risky to human beings. The exposure of heavy metals happens through anthropogenic events such as smelting and mining operations, domestic and other industrial products and lastly, through the use of metal and its related components in the agricultural industry, [5,6].

Volcanic eruption and weathering are examples of a few of the natural occurrence which contributes to the exposure of heavy metals [5,7,8].

2.3 Zinc in the environment

A common trace element, Zinc is indispensable and is needed by the body, it is also an essential trace element needed by microorganisms, animals and plants, for the evolution and development of the microorganisms [9]. Historically, the sources of heavy metals to sludge have been industrial activities such as surface treatment with elements such as Cu, Zn, Ni and Cr. In recent years, industries have to have a large degree moved out of the cities and the release of heavy metals and other compounds has decreased due to various pre-treatments of the effluent. This is reflected in the total amount of heavy metals in sludge from WTP [10]. On the other hand, zinc can also have adverse reactions on environment because it can cause several problems when released in very small quantities, since its toxicity grows by bioaccumulation.

2.4 Conventional technologies for heavy metals removal

Different methods such as ion exchange, chemical precipitation, membrane filtration, coagulation and flocculation, electrochemical treatment and adsorption are mostly used to remove heavy metal from wastewater.

2.4.1 Ion Exchange

Ion-exchange resin, both natural solid or synthetic resins, has a unique capability to interchange its cations with the heavy metals in wastewater. When used as material in ion-exchange methods, synthetic resins are preferred as they are more effective in removing the metal ions from the wastewater [13]. A number of research has found that zeolites shows high capacities to metal ions cation-exchange under a wide spectrum of investigational conditions [14,15].

2.4.2 Chemical Precipitation

When dealing with inorganic effluents, chemical precipitation is a method used commonly to remove heavy metals. The most accessible and commonly used precipitant agents are limestone and lime. Limestone and lime cheap and can be found anywhere [16]. However, chemical precipitation causes poor settling, slow precipitation of metal, production of excess sludge which needs additional treatment, long term sludge disposal impacts and high accumulation of metal precipitates [16].

2.4.3 Filtration using Membrane

Membrane filtration and with the use of other membrane is an excellent technique as it is easy to operate, saves space and has a high efficiency rate.

2.5 Adsorption

The most economic and effective technique for the treatment of wastewater which contains heavy metals currently is adsorption. This is because, the adsorption technique is flexible and produces better quality results. Moreover, adsorption is a reversible process, thus, it is able to regenerate the adsorbent.

2.6 Adsorbents from activated carbon

In treating wastewater contaminated by heavy metals, activated carbon is usually chosen in the adsorption technique. The large mesopores and micropore volumes and large surface area contributes to the success when using activated carbon as an adsorbent [17]. In addition to this technique, an advancement to the technique must be made by using activated carbon composite and additives without the declining expense of the contaminants adsorption.

2.7 Modified activated carbon for the removal of zinc from wastewater

Modified activated carbon are carbonaceous adsorbents which have tetra butyl ammonium iodide (TBAI) and sodium diethyl dithiocarbonate (SDDC) immobilised at their surface. This study investigates the adsorption of toxic ions, copper, zinc, chromium and cyanide on these adsorbents that have undergone surface modification with tetra butyl ammonium (TBA) and SDDC in wastewater applications. The modification technique enhances the removal capacity of carbon and therefore decreases cost-effective removal of Zn(II) from metal finishing (electroplating unit) wastewater. Two separate fixed bed modified activated carbon columns were used TBA-carbon column for cyanide removal and SDDC-carbon column for metal ions Zn removal. Wastewater from electroplating unit containing Zn 9.5 mg was treated through the modified columns. [18]

2.8 Adsorption of zinc from aqueous solutions by using natural clay

In this study, removal of Zinc (Zn2+) from aqueous solutions is investigated using Cankırı bentonite, a natural clay. During the removal process, batch technique is used, and the effects of pH, clay amount, heavy metal concentration and agitation time on adsorption efficiency are studied. Langmuir, Freundlich and Dubinin–Radushkevich (D–R) isotherms are applied in order to determine the efficiency of natural clay used as an adsorbent. Results show that all isotherms are linear. It is determined that adsorption of Zn (II) is well-fitted by the second order reaction kinetic. In addition, calculated and experimental heavy metal amounts adsorbed by the unit clay mass are too close to each other. It is concluded that natural clay can be used as an effective adsorbent for removing Zn (II) from aqueous solutions. [19]

2.9 Removal of Zinc from Aqueous Solutions Using Bagasse Fly Ash

Bagasse fly ash, a sugar industry waste, has been converted into an inexpensive and efficient adsorbent. The product obtained has been characterized and utilized for the removal of zinc from aqueous solutions over a wide range of initial metal ion concentration (3.06×10^{-4} to 3.06×10^{-3}M), contact time (24 h), adsorbent dose (5−20 g L^{-1}), and pH (1.0−6.0). The removal of Zn^{2+} is 100% at low concentrations, whereas it is 60−65% at higher concentrations at an optimum pH of 4.0, using 10 g L^{-1} of adsorbent in 6−8 h of equilibration time. The uptake decreases with a rise in temperature indicating the process to be exothermic in nature. Kinetic studies have been performed to understand the mechanism of adsorption.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH STUDY FLOWCHART

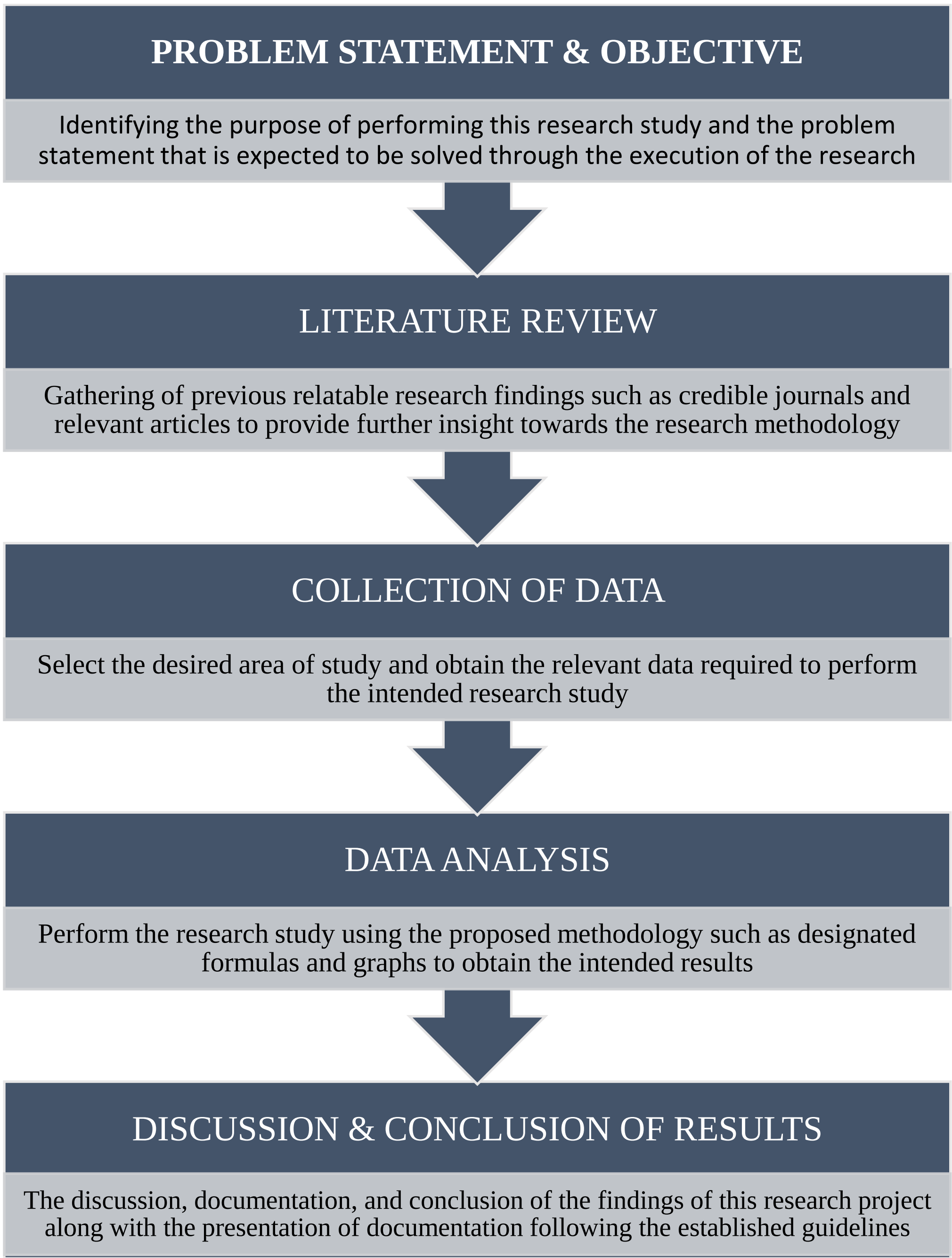

FIGURE 4 : Project Framework

The topic adsorption of heavy metals is not fairly new, as it has been studied many times before by various researchers. The study approach and the methodology used uses the concept of experimental and modelling interface. However, a new method is tried out for this topic, which uses dry activated sludge to obtain the activated carbon. Based on the research and study made, using dry activated sludge is uncommon amongst researchers as most researches uses the more alternative and conventional ways. As the aim of this FYP topic is to treat the wastewater in order to reduce the amount of landfill, the usage of dry sludge is recycled and uses fully covered and partially covered wastewater treatment to remove the heavy metals. The past journal and research paper studies on the experimental procedures and methodology used for this experiment is referred to for this topic to ensure the project meets the expected outcome.

This FYP project is done in stages and within a duration of 8 months. Throughout the duration, certain modifications and improvement were added to the experiment to achieve the desired result. Inputs and ideas by supervisor, and also through reading of journal papers are taken into account to ensure the validity and better result from the experiment.

3.2 MATERIAL AND EXPERIMENT METHODOLOGY

3.2.1 Production of Activated Carbon

The sludge is obtained from the Sewage Treatment Plant (UTP)'s drying bed. From the sludge obtained, it is then used to produce the needed activated carbon. The sludge is dried in an oven at for 24 hours at a temperature of 105°C until the sludge loses all its moisture and the desired constant weight of 30g is achieved. The 30g of dried sludge is then proceeded to be incinerated in a tube furnace for three hours with a constant temperature of 500 °C. During the production process, the heating rate is maintained at an increase 10 °C/min with a nitrogen flow rate of about one hundred millilitre (100 mL/min). Lastly, the obtained sludge activated carbon is manually grinded to an average size of 5 cm and crushed and sieved into a powder form of about 150 µm. The sludge activated carbon (SAC) powder is stored in a desiccator prior to its usage.

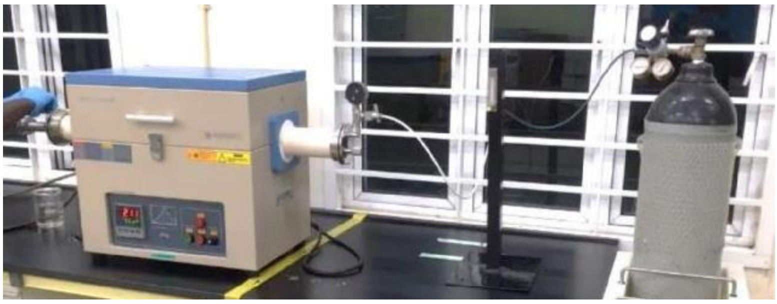

FIGURE 5: Furnace for pyrolysis

FIGURE 6: Initial dried sludge directly carbonized by pyrolysis at 500 °C

3.2.2 Experimental procedures

Batch adsorption experiments is carried out with the obtained SAC as the adsorbent. By using 30 pieces of 250 mL Erlenmeyer flask, a hundred millilitre (100 mL) synthetic zinc (ZnII) residue which has a fixed metal ion concentration is placed in the series of flasks. Then, 0.5, 1.0, 2.5, 5.0, 7.5 and 10.0 g/L of SAC is also placed in the Erlenmeyer flasks accordingly. An orbital shaker (Protech Model 722) is set at a speed of 250 rpm at room temperature (25 ± 1°C). The flasks are then agitated using the orbital shaker with the set settings. Using the Whatman's glass microfiber filters paper (GF/C), the mixtures are then separated at the end of its scheduled agitation times of 3, 6, 8, 12 and 24 hours. Filtered solutions were analysed for residual zinc (II) concentration using a spectrophotometer (DR5000 Hach). The tests were run in triplicates at three different zinc metal concentrations of 10 mg/L, 50 mg/L and 100 mg/L. At the end of the predetermined interval of time, samples were removed from the shaker and analysed for residual zinc concentration until equilibrium was attained.

FIGURE 7: Mechanical orbital shaker

FIGURE 8: Spectrophotometer (DR 5000 Hach)

Equations (1) and (2) shown below are used in computing the adsorption efficiency of the metal and the adsorption capacity of the SAC.

$$R = \frac{(Ci-Ce)}{Ci} * 100 \quad (1)$$

Equations (1) and (2) shown below are used in computing the adsorption efficiency of the metal and the adsorption capacity of the SAC.

$$Q_c = \frac{(Ci-Ce)}{W} \, V \quad (2)$$

Referring to equation (2), the adsorbent adsorption capacity (mg/g) is denoted as Qc, and the parameter V is the volume of aqueous Zinc solution (L) and W is the weight of the SAC (g).

FIGURE 9: Whatman’s glass microfiber filters paper (GF/C)

3.2.3 Adsorption isotherm studies (performance of SAC)

The adsorption isotherm studies establishes the connection between adsorbate concentration and the degree of adsorption which explains the amount of adsorbent adsorbed onto the surface of the adsorbent. In the same study referred, the models used to measure the adsorption capacity of SAC for removal of zinc from aqueous solution are the Freundlich and Langmuir isotherm models. Freundlich isotherm model is a model commonly used to describe adsorption equilibrium based on an empirical equation. By using the Freundlich isotherm model, we are able to understand the relationship of adsorption related to both organic and inorganic composites on a wider range of adsorbents including biosorbents. The linearized equation for the Freundlich isotherm model is:

$$\log A_q = \log K_F + \frac{1}{n} \log C_e \qquad (3)$$

where log A_q is the quantity of the adsorbed pollutant per gram of the adsorbent (mg/g), C_e is the equilibrium concentration (mg/L), K_F and n are Freundlich constants. Plot of $\mathbf{\log A_q}$ against $\mathbf{\log C_e}$ has $\mathbf{\frac{1}{n}}$ as slope and $\mathbf{\log K_F}$ as its intercept.

Another frequently used model is the Langmuir model which is used to describe sorption of heavy metals unto biosorbents. Langmuir model is used based on three well-defined assumptions ; that all surface sites are the same and can occupy one adsorbed atom, it is limited to monolayer coverage adsorption and capability of a molecule adsorbed at a given site is not dependent on the occupancy of its adjoining sites. The Langmuir isotherm model equation is as follows:

$$\frac{C_e}{A_q} = \frac{1}{b_L\, q_m} + \frac{C_e}{q_m} \quad (4)$$

Referring to equation (4) above, Aq is the quantity of pollutant adsorbed per gram of adsorbent (mg/g), bL is the Langmuir constant for equilibrium, qm is the adsorbate quantity that is required for a monolayer formation and Ce is the concentration of residual metal at equilibrium (mg/L). Thus, plot of $\frac{C_e}{A_q}$ against $\boldsymbol{Ce}$ should produce a straight line with a slope of $\frac{1}{q_m}$ and $\frac{1}{b_{Lqm}}$ as the intercept if the adsorption obeys the Langmuir equation [31]. This important characteristic of the Langmuir isotherm can be expressed in terms of a dimensionless factor, RL [44], which is defined as:

$$R_L = \frac{1}{1+b_L\, q_m} \qquad (5)$$

The RL values indicate the type of adsorption, whether it is unfavourable ($RL > 1$), linear ($RL = 1$), favourable ($0 < RL < 1$), or irreversible ($RL = 0$).

3.2.4 Pseudo-first and second order kinetic models

Kinetics of zinc adsorption using SAC has also been studied by various past researchers. Lagergren first and second order kinetic models were also utilized for this study. Expression of Pseudo first order kinetic model is expressed generally as follows.

$$\log q_{eq} - \frac{k_1}{2.303} t \quad (6)$$

where q_{eq} is the quantity of adsorbate adsorbed at equilibrium (mg/g), qt is the quantity adsorbate adsorbed at a given time t and $k1$ is the first order constant rate of adsorption (min^{-1}). Plot of log (q_{eq}- qt) against time should be linear with $\frac{K_1}{2.303}$ and log q_e representing the slope and intercept of the line.

$$\frac{t}{q_t} = \frac{1}{k_{2\ q_{eq}^2}} + \frac{1}{q_{eq}} (\mathrm{t}) \qquad (7)$$

where k_2 is the second order adsorption rate constant (g $\mathrm{mg}^{-1}\mathrm{min}^{-1}$) and q_{eq} is the adsorbed quantity of pollutant at equilibrium (mg/g). The second order plots of $\frac{t}{qt}$ against time will yield straight.

TASKS	FYP 1														FYP 2													
Week	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**
FYP Topic																												
Selection of project title and meeting with FYP Coordinator	■	■	■																									
Data Gathering and Analysis																												
Preliminary Research Work (Reference)		■	■	■	■	■	■	■	■	■	■	■	■	■														
Organizing literature review									■	■	■	■	■	■														
Extended Proposal																												
Proposal Defence										■	■																	
Project Activities																												
Carbon Activation												■	■	■														
Heavy Metal Adsorptions															■	■	■	■	■	■	■							
Interim Report																												
Preparation & Submission													■	■														
Progress Report																												
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CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Adsorption Experiment

Zinc (II) Chloride solution is used to conduct the activated carbon reaction. The zinc removal was determined based on two effects such as:

i. Effect of contact time and initial metals ion concentration

ii. Effect of adsorbent dosage and initial metals ion concentration.

4.1.1 Effect of contact time

The Figure 10 below shows the adsorption of zinc at a concentration of 10 mg/L which the reaction occurs between 12 to 24 hours of interval time. The graph for the adsorbent dosage vs contact time shows a similar constant pattern for all the dosages between except for the dosage 10 g/l in which it remains at equilibrium state between 8 to 12 hours. The removal efficiency between 12 to 24 hours is the highest increase. At the end of 24 hours interval, it can be observed that the pores in the adsorbent were fully adsorbed causing significant changes in the removal and adsorption process was met.

Adsorbent dosages (g/l)	Contact Time (hours)				
	3	**6**	**8**	**12**	**24**
0.5	4.72	3.85	3.69	3.76	3.62
1.0	4.31	4.11	3.92	3.83	3.54
2.5	4.13	3.87	3.76	3.68	3.35
5.0	3.97	3.78	3.72	3.67	3.55
7.5	3.48	3.32	3.23	3.07	2.86
10.0	3.58	3.42	3.04	2.84	2.44

TABLE 1: Zn(II) at 10 mg/L initial metals ion concentration and various contact time

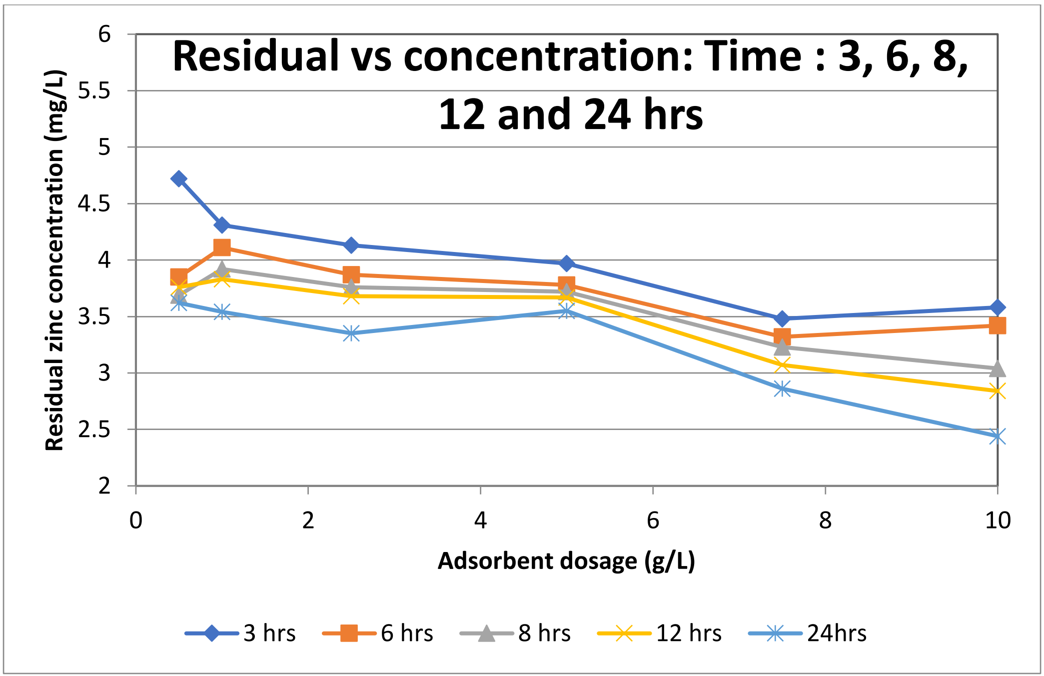


FIGURE 10: Zinc Residual (10 mg/L) vs Contact Time

Adsorbent dosages (g/l)	**% Removal efficiency**				
	3	**6**	**8**	**12**	**24**
0.5	52.8	61.5	63.1	62.4	63.8
1.0	56.9	58.9	60.8	61.7	64.6
2.5	58.7	61.3	62.4	63.2	66.5
5.0	60.3	62.2	62.8	63.3	64.5
7.5	65.2	66.8	67.7	69.3	71.4
10.0	64.2	65.8	69.6	71.6	75.6

TABLE 2: Removal efficiency (%) of Zn(II) at 10 mg/L initial metals ion concentration and various contact time

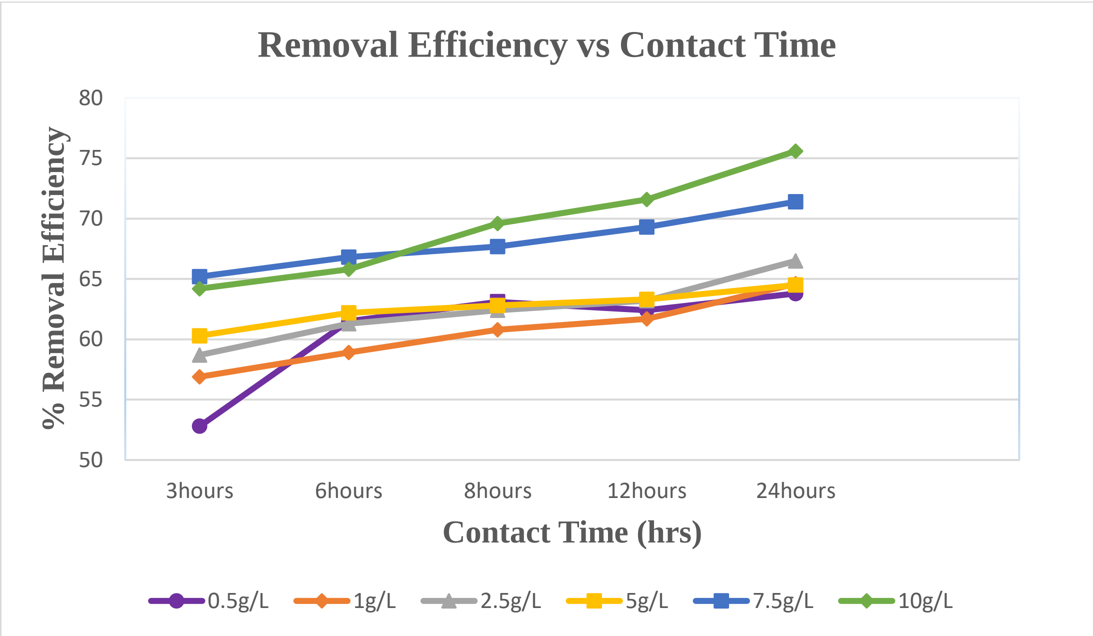


FIGURE 11: Removal Efficiency vs Contact Time (10mg/L)

The Figure 12 below shows the adsorption of zinc at a concentration of 50 mg/L which the reaction occurs between 8 to 12 hours of interval time. The graph for the zinc residual vs contact time shows a similar constant pattern for all the dosages in which it remains at equilibrium state between 12 to 24 hours. The removal efficiency between 12 to 24 hours are the highest increase. At the end of 24 hours interval, it can be observed that the pores in the adsorbent were fully adsorbed causing significant changes in the removal and adsorption process was met.

Adsorbent dosages (g/l)	**Contact Time (hours)**				
	3	**6**	**8**	**12**	**24**
0.5	3.55	3.5	3.18	2.33	2.08
1.0	2.99	2.58	2.51	2.13	1.46
2.5	1.75	1.34	1.23	1.09	0.55
5.0	0.79	0.6	0.55	0.44	0.25
7.5	0.66	0.6	0.32	0.25	0.16
10.0	0.51	0.48	0.22	0.18	0.13

TABLE 3: Zn(II) at 50 mg/L initial metals ion concentration and various contact time

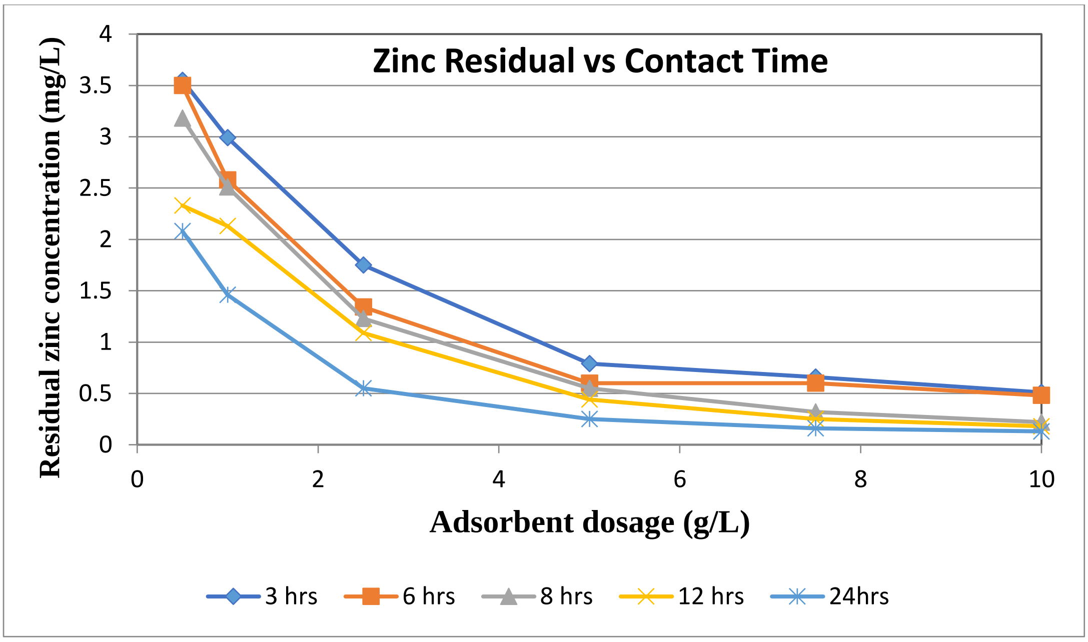


FIGURE 12: Zinc Residual (50 mg/L) vs Contact Time

Adsorbent dosages (g/l)	**% Removal efficiency**				
	3	**6**	**8**	**12**	**24**
0.5	92.9	93.0	93.6	95.3	95.8
1.0	94.0	94.8	95.0	95.7	97.0
2.5	96.5	97.3	97.5	97.8	98.9
5.0	98.4	98.8	98.9	99.1	99.5
7.5	98.7	98.8	99.4	99.5	99.7
10.0	98.9	99.0	99.6	99.65	99.7

TABLE 4: Removal efficiency (%) of Zn(II) at 50 mg/L initial metals ion concentration and various contact time

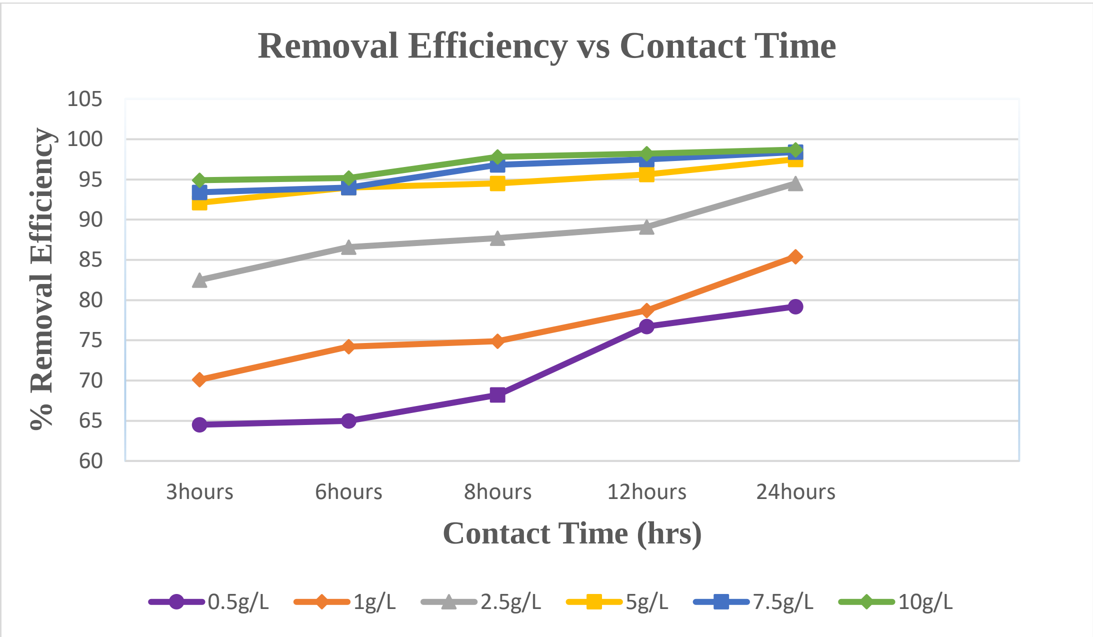


FIGURE 13: Removal Efficiency vs Contact Time (50mg/L)

The Figure 14 below shows the adsorption of zinc at a concentration of 100 mg/L which the reaction occurs between 6 to 8 hours of interval time. The graph for the zinc residual vs contact time shows a similar constant pattern for all the dosages in which it remains at equilibrium state between 8 to 12 hours. The removal efficiency between 12 to 24 hours is the highest increase. At the end of 24 hours interval, it can be observed that the pores in the adsorbent were fully adsorbed causing significant changes in the removal and adsorption process was met.

Adsorbent dosages (g/l)	Contact Time (hours)				
	3	6	8	12	24
0.5	4.37	4.32	4.18	4.07	3.38
1.0	4.29	4.25	3.93	3.85	3.72
2.5	4.18	4.13	3.77	3.70	3.38
5.0	4.12	4.08	3.63	3.50	3.28
7.5	3.89	3.80	3.54	3.35	3.16
10.0	3.88	3.76	3.38	3.12	2.85

TABLE 5: Zn(II) at 100 mg/L initial metals ion concentration and various contact time

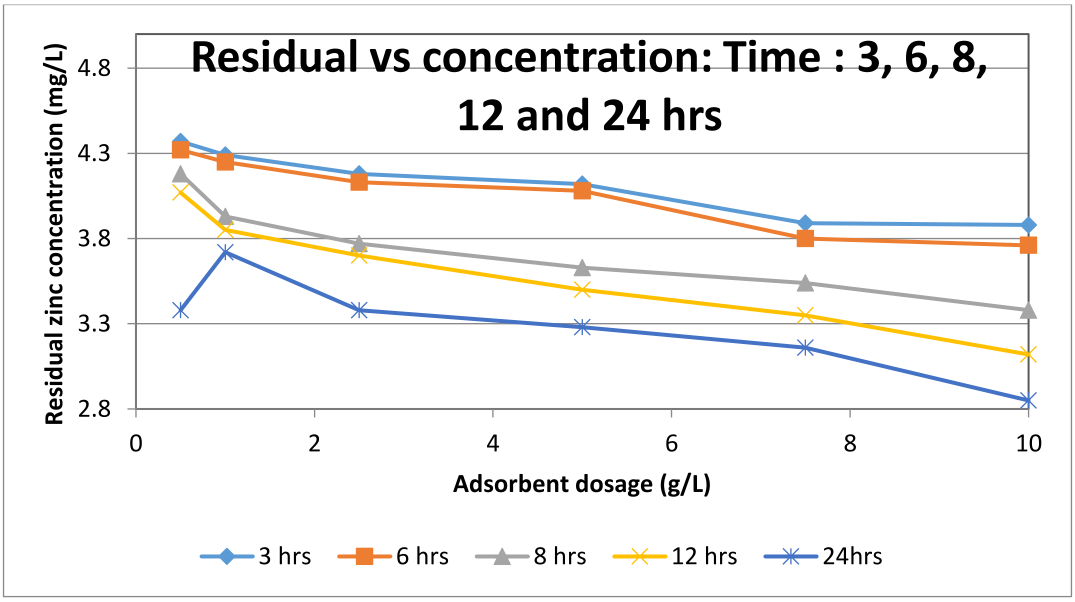


FIGURE 14: Zinc Residual (100 mg/L) vs Contact Time

Adsorbent dosages (g/l)	**% Removal efficiency**				
	3	**6**	**8**	**12**	**24**
0.5	95.6	95.7	95.8	95.9	96.6
1.0	95.7	95.8	96.1	96.2	96.3
2.5	95.8	95.9	96.2	96.3	96.6
5.0	95.9	95.9	96.4	96.5	96.7
7.5	96.1	96.2	96.5	96.7	96.8
10.0	96.1	96.2	96.6	96.9	97.2

TABLE 6: Removal efficiency (%) of Zn(II) at 100 mg/L initial metals ion concentration and various contact time

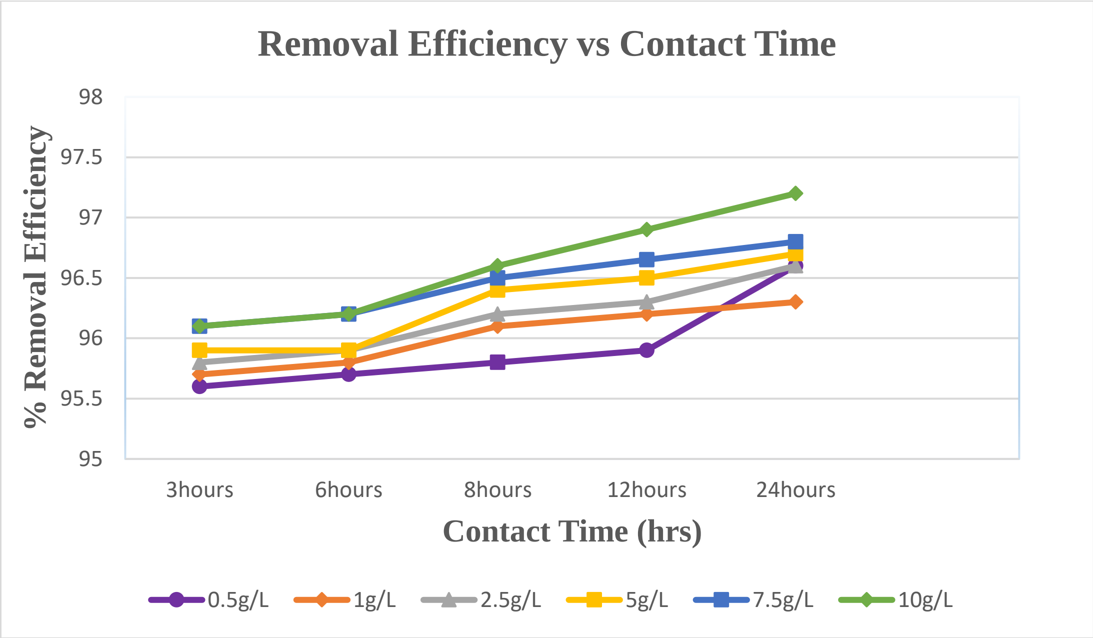


FIGURE 15: Removal Efficiency vs Contact Time (100mg/L)

4.1.2 Effect of adsorbent dosage

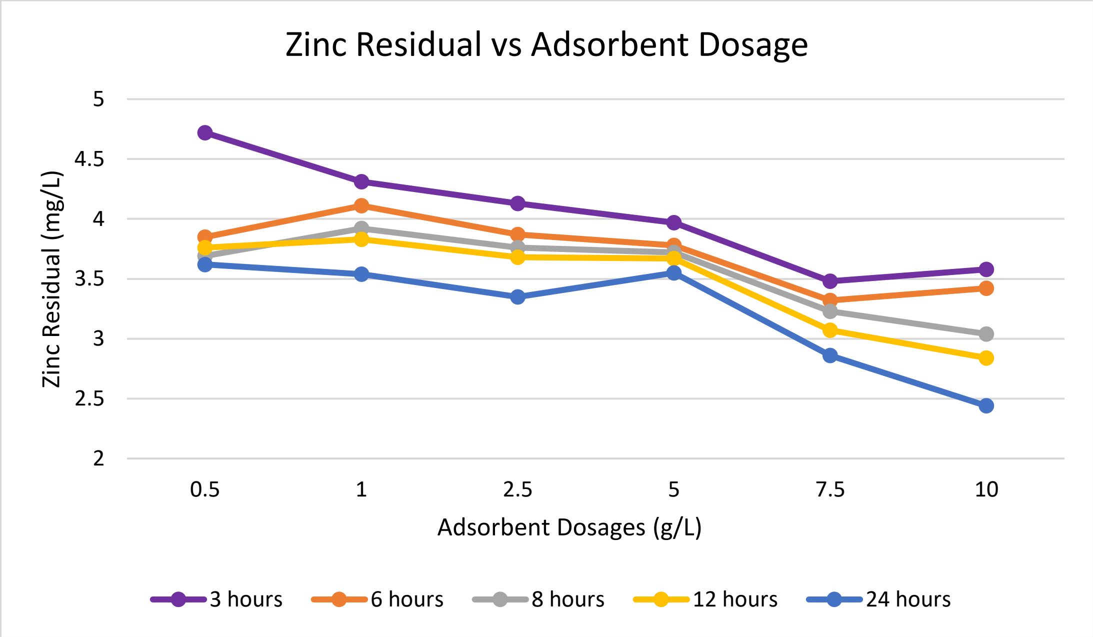


FIGURE 16: Zinc Residual (10 mg/L) vs Adsorbent Dosage

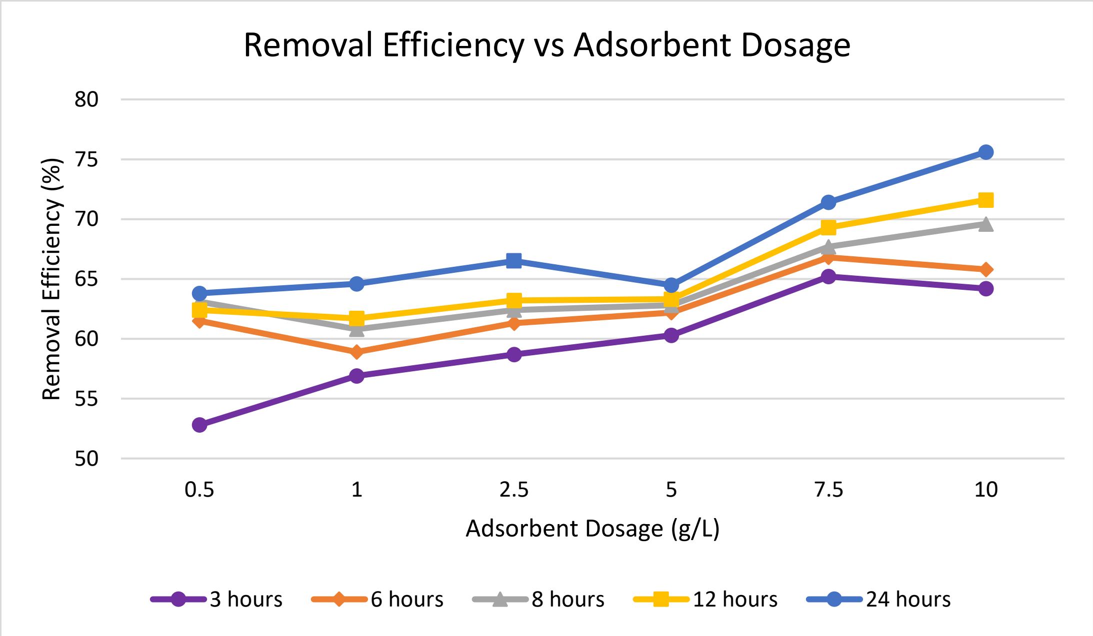


FIGURE 17: Removal Efficiency vs Adsorbent Dosage (10mg/L)

Figure 16 and 17 indicates the reaction of different dosages of SAC for the removal efficiency of zinc metal. The removal efficiency of zinc metal ion can be observed by determined the increase of zinc metal ions (reading shown was in unstable condition due to problems faced during experiment was conducted) with the increasing of adsorbent dosage. Besides that, there was a constant and insignificant changes occur at the interval time of 6, 8,12 and 24 hours which the removal efficiency has reached 70%-75%. This indicates that the increase of SAC dosages causing the removal efficiency of zinc metal to increase hence more sites are available to bind. However, it can be concluded that adsorption process occurred at the interval time of 24 hours and the uptake capacity of zinc metal ions per gram of SAC was reduced with the effect of increase in SAC dosages.

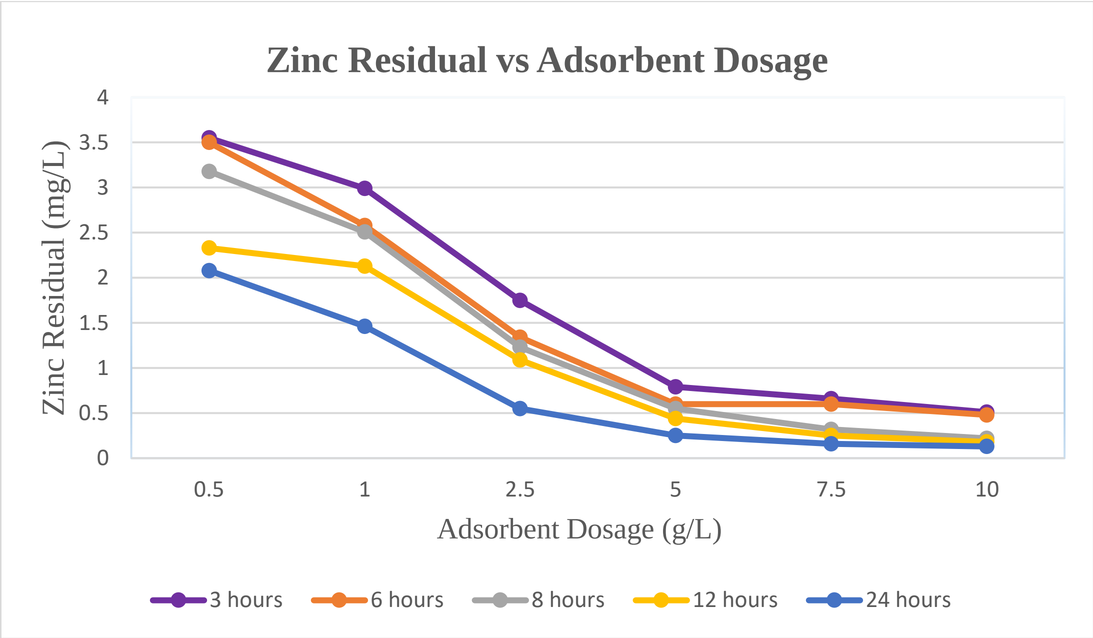


FIGURE 18: Zinc Residual (50mg/L) vs Adsorbent Dosage

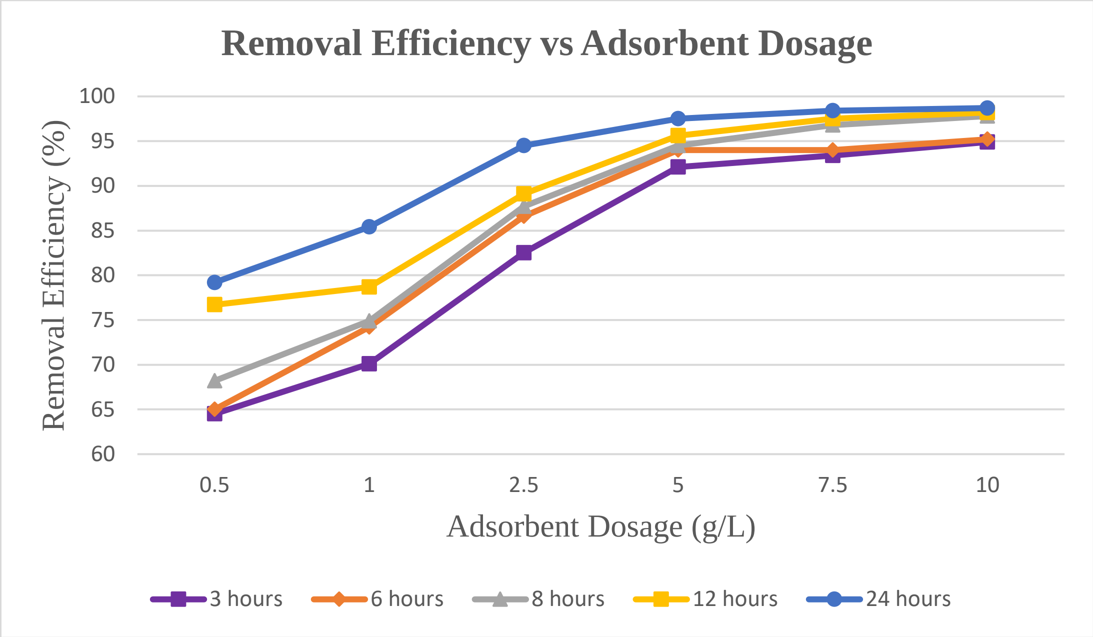


FIGURE 19: Removal Efficiency vs Adsorbent Dosage (50mg/L)

Figure 18 and 19 above shows same observation occur to SAC with zinc concentration of 50 mg/L which the removal efficiency of Zn(II) adsorption by SAC was increased as well as the adsorbent dosages but after certain period it become slightly constant and reading showed insignificant changes which the SAC dosages has reached 7.5-10.0g/L for the zinc metal adsorption process. 95%-100% of zinc removal efficiency shown at the SAC dosages between 7.5g/L to 10g/L. Therefore, the equilibrium state and optimum condition of adsorbent dosage was reached at 10g/L of SAC dosage.

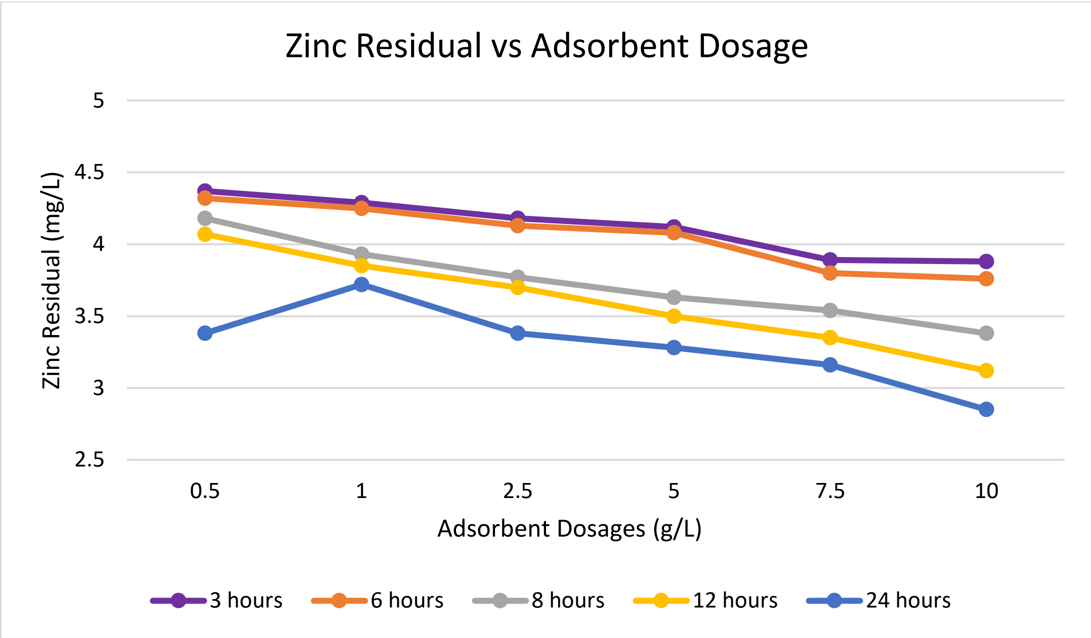


FIGURE 20: Zinc Residual (100mg/L) vs Adsorbent Dosage

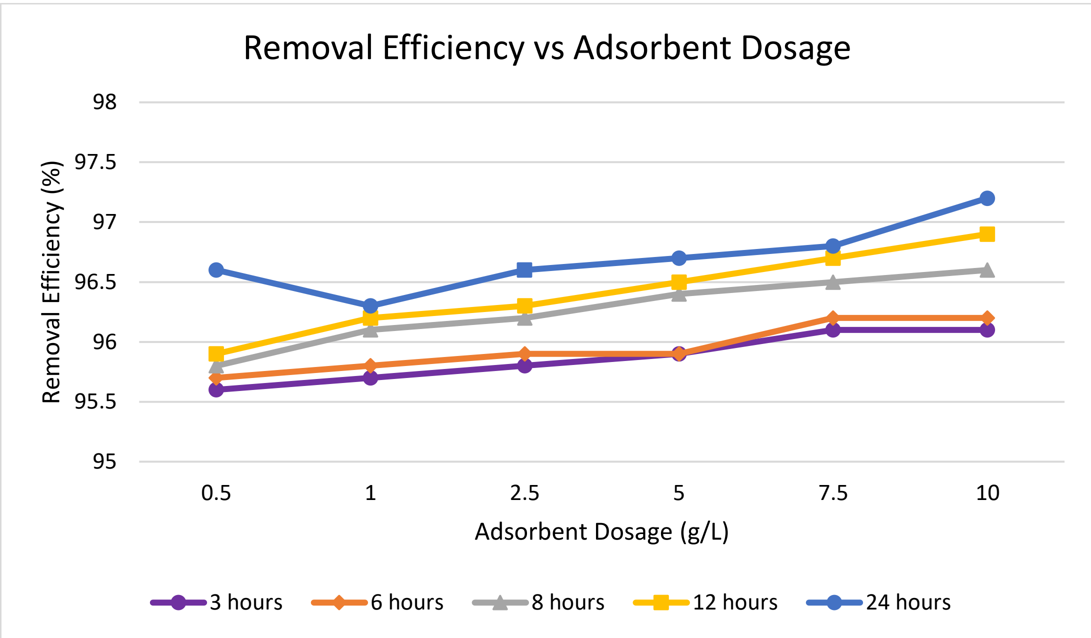


FIGURE 21: Removal Efficiency vs Adsorbent Dosage (100mg/L)

Figure 20 and 21 above shows same observation occur to SAC with zinc concentration of 100 mg/L which the removal efficiency of Zn(II) adsorption by SAC was increased as well as the adsorbent dosages but after certain period it become slightly constant and reading showed insignificant changes which the SAC dosages has reached 7.5g/L for the zinc metal adsorption process. 95%-97% of zinc removal efficiency shown at the SAC dosages between 7.5g/L to 10g/L. Therefore, the equilibrium state and optimum condition of adsorbent dosage was reached at 7.5g/L of SAC dosage which the graph of 24 hours contact time is exceptional.

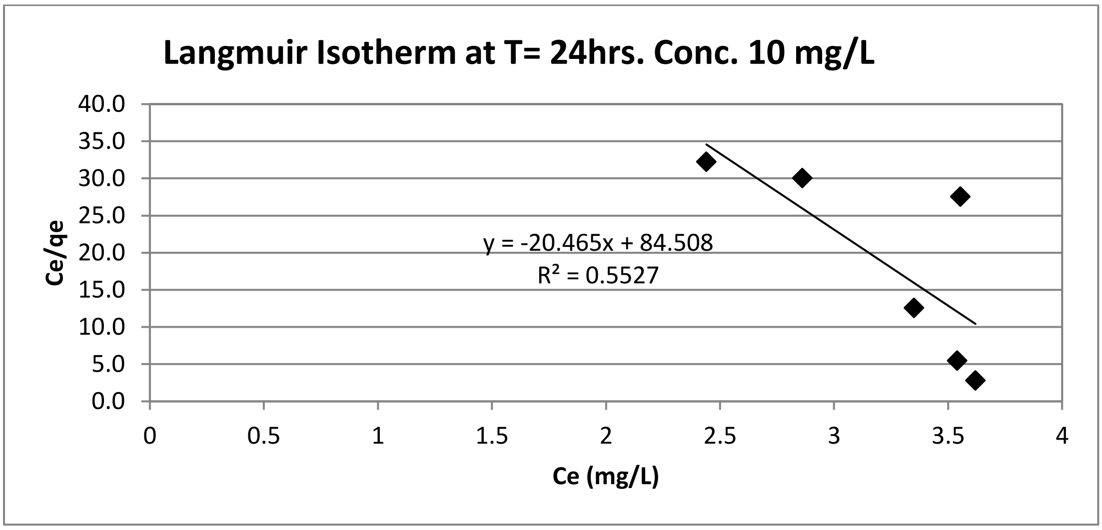
4.2 Adsorption Isotherm

The adsorbents and the zinc metal ions reactions can be determined and is decisive to optimize the bio-adsorbents utilization [18]. The Freundlich isotherm model defined that the adsorption occur at the multiple layer and heterogeneous surfaces of adsorbent which binding sites occur was not equivalent. The interaction of adsorbed solute particles were repulsive (Freundlich, 1906; Sinha et al., 2012). Freundlich isotherm parameters can be determined by using the Freundlich equation which can be linearized on logarithmic form for graph plotting.

While the Langmuir isotherm model defined that monolayers occur the adsorbent during adsorption process which contain numbers of identical sites. The interaction of adsorbate particles was negligible for this scenario. Linear form of Langmuir isotherm adsorption is analysed in the equilibrium reading which indicates the monolayer that occurs in the biosorption capacity which was calculated from the slope and interception point of the plot versus Ce.

Referring to Figure 22, the two graphs plot shown are the Freundlich and Langmuir isotherm plot. The Freundlich plot shows the plot of log Qe against log Ce. The two points in Freundlich isotherm is located on the positive value of log Qe which indicates that the adsorption of the zinc metal is successful. Therefore, from the plot of Freundlich isotherm, it can be concluded that the Freundlich isotherm model best matches the result of this experiment rather than Langmuir isotherm model. Lastly, small heterogenous adsorption patches makes up the SAC surface which is in line with the Freundlich type adsorption which explains heterogeneity of the adsorbent. [19]

The R^2 values in the plot shows a value of less than 1 which explains that the adsorption of zinc metal is highly favourable. As the adsorbent dosages increases, the zinc metal adsorption also increases. This means there will be significant increment in heavy metal adsorption with further increase of adsorbents dosage. Comparing the two isotherm plots, the Freundlich isotherm plot has a higher value than the Langmuir isotherm plot. This indicates that zinc metal ions by SAC adsorbent has the maximum uptake. The reading shows that zinc metals removal is higher with SAC.



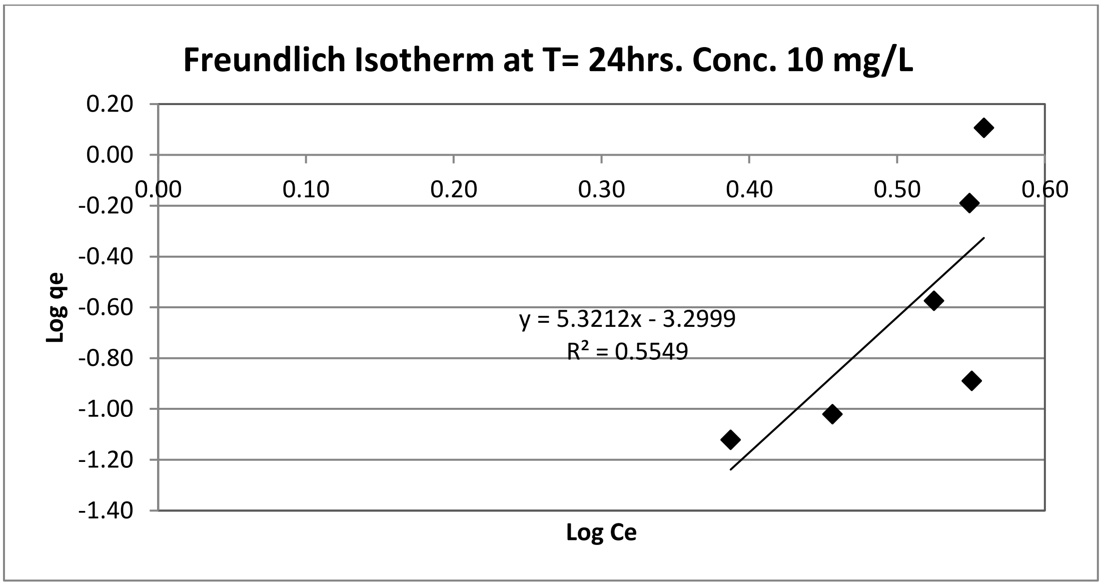
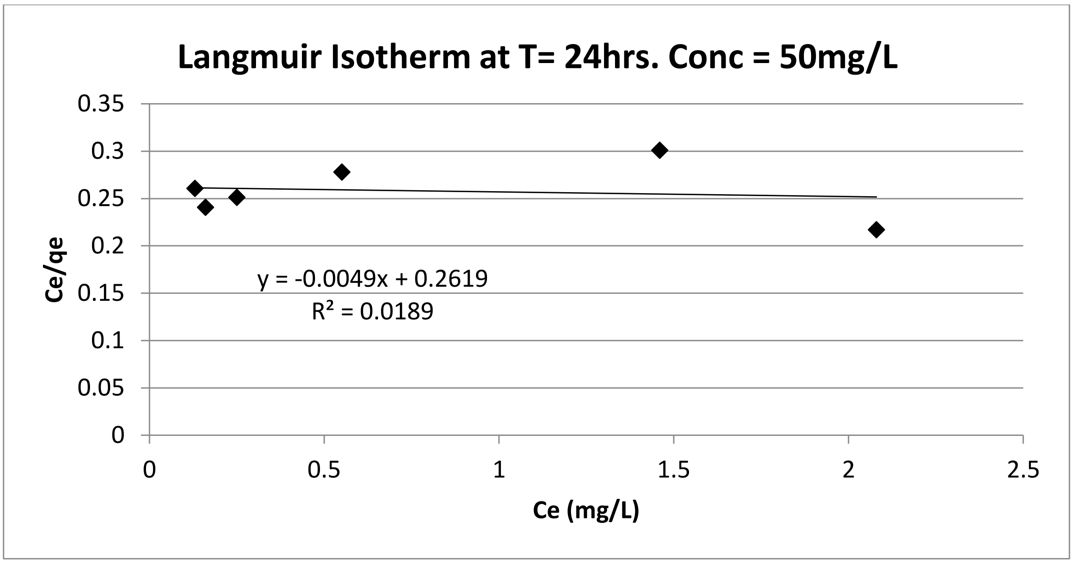


FIGURE 22: Freundlich and Langmuir of Zn (**10mg/L**)



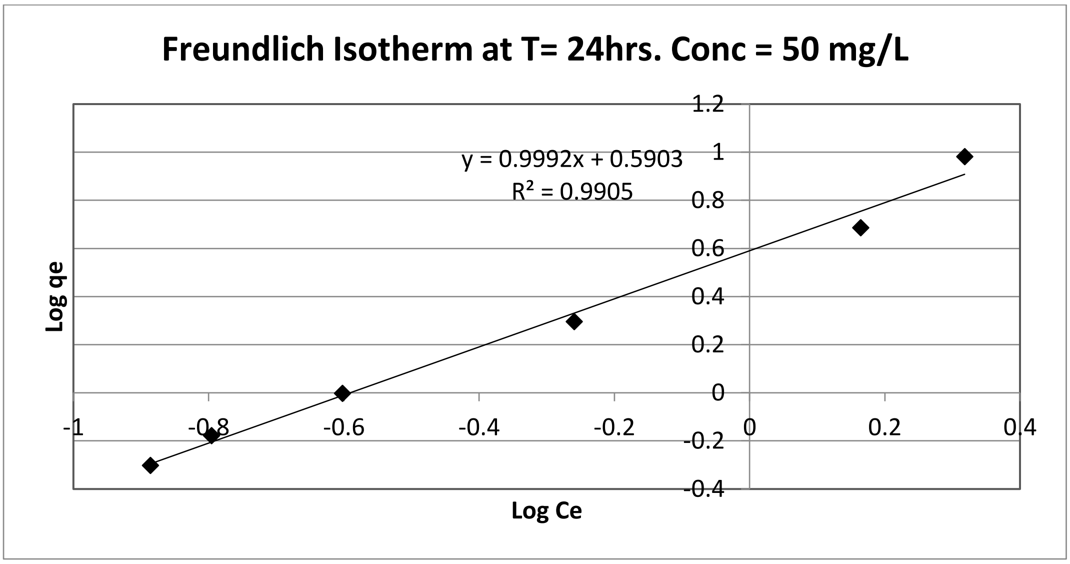
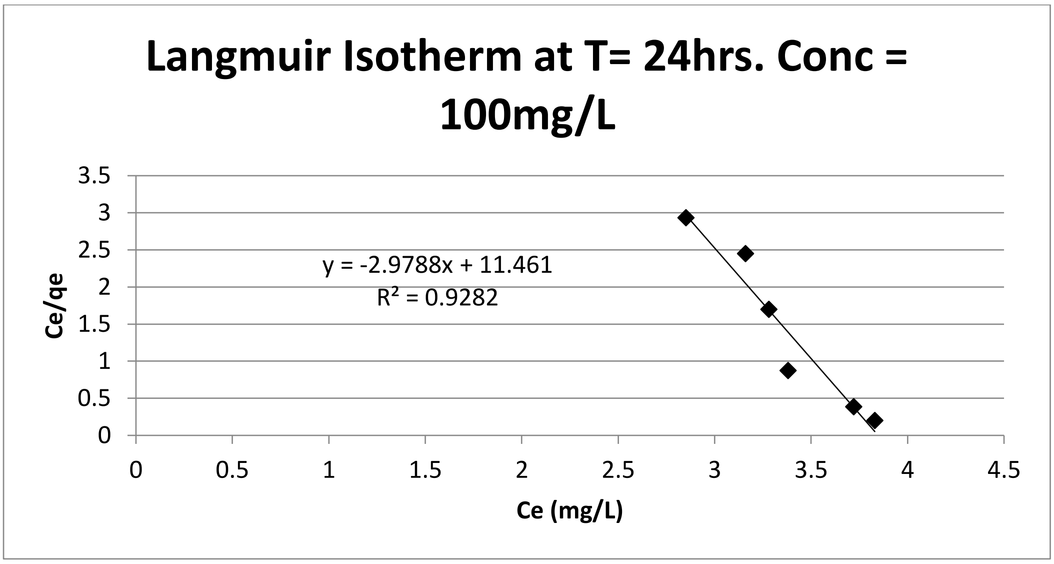


FIGURE 23: Freundlich and Langmuir of Zn (**50mg/L**)

Referring to Figure 23, the two graphs plot shown are the Freundlich and Langmuir isotherm plot. From the plot the Freundlich isotherm parameters are completed from the intercept and gradient of the linear plot. The Freundlich plot shows the plot of log Qe against log Ce. The two points in Freundlich isotherm is located on the positive value of log Qe which indicates that the adsorption of the zinc metal is successful. Therefore, from the plot of Freundlich isotherm, it can be concluded that the Freundlich isotherm model best matches the result of this experiment rather than Langmuir isotherm model. Lastly, small heterogenous adsorption patches makes up the SAC surface which is in line with the Freundlich type adsorption which explains heterogeneity of the adsorbent. [19]

The R^2 values in the plot shows a value of less than 1 which explains that the adsorption of zinc metal is highly favourable. As the adsorbent dosages increases, the zinc metal adsorption also increases. This means there will be significant increment in heavy metal adsorption with further increase of adsorbents dosage. Comparing the two isotherm plots, the Freundlich isotherm plot has a higher value than the Langmuir isotherm plot. This indicates that zinc metal ions by SAC adsorbent has the maximum uptake. The reading shows that zinc metals removal is higher with SAC.



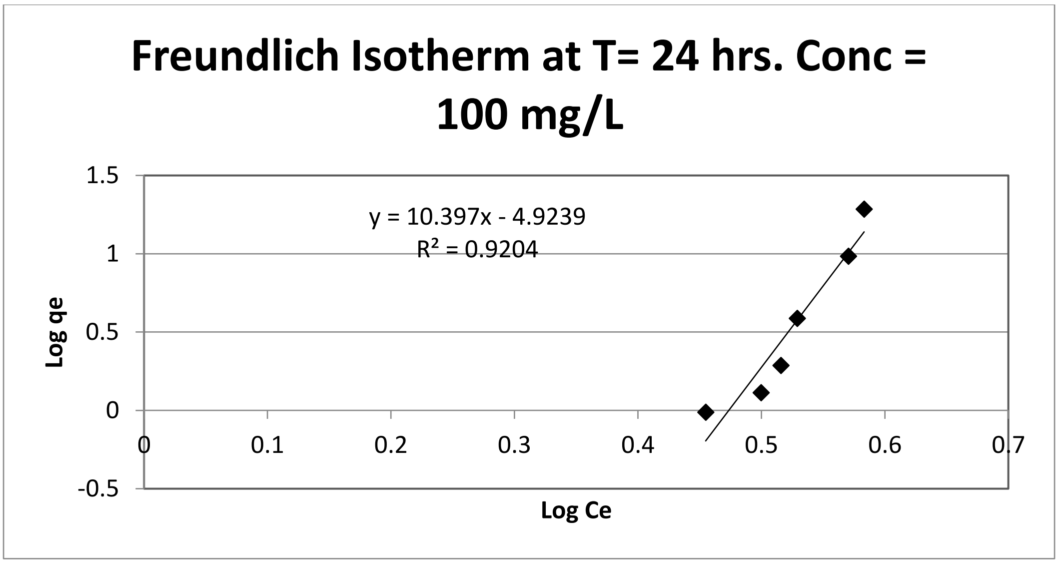
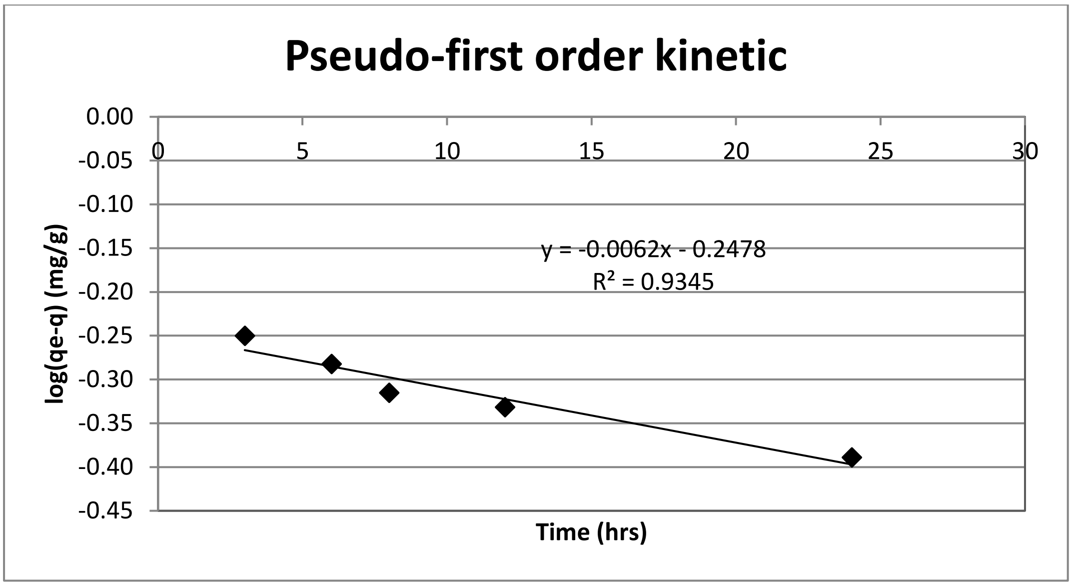


FIGURE 24: Freundlich and Langmuir of Zn (**100mg/L**)

Referring to Figure 24, the two graphs plot shown are the Freundlich and Langmuir isotherm plot. From the plot the Freundlich isotherm parameters are completed from the intercept and gradient of the linear plot. The Freundlich plot shows the plot of log Qe against log Ce. The two points in Freundlich isotherm is located on the positive value of log Qe which indicates that the adsorption of the zinc metal is successful. Therefore, from the plot of Freundlich isotherm, it can be concluded that the Freundlich isotherm model best matches the result of this experiment rather than Langmuir isotherm model. Lastly, small heterogenous adsorption patches makes up the SAC surface which is in line with the Freundlich type adsorption which explains heterogeneity of the adsorbent. [19]

4.3 Kinetic Study



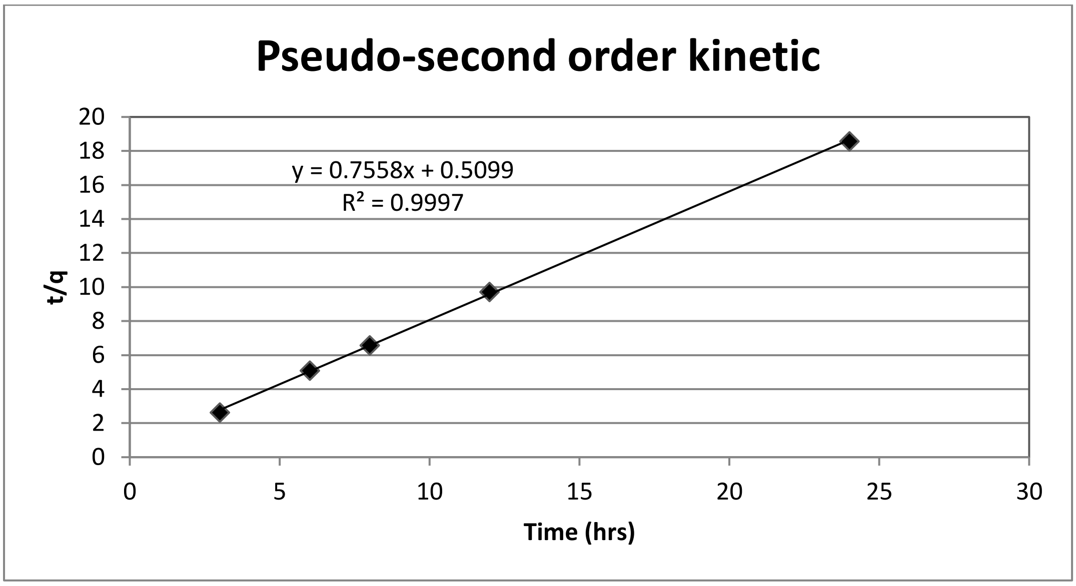
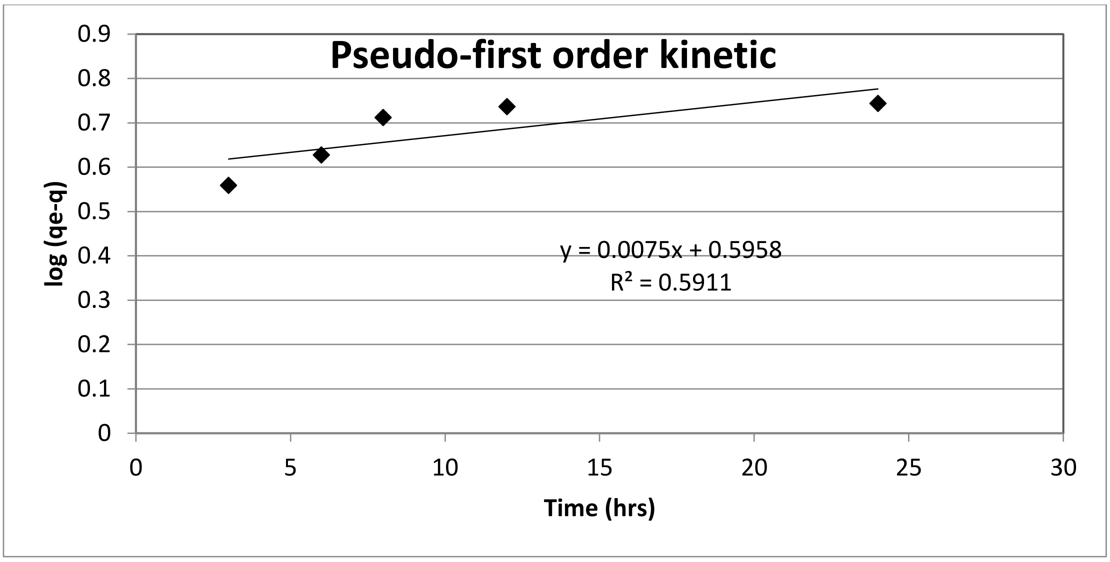


FIGURE 25: Pseudo-first & second order of **10 mg/L**

Referring to Figure 25 above, the data indicates that the pseudo-first order kinetic model has a lower value compared to pseudo-second order kinetic. Thus, the result from this experiment obeys the pseudo-second order kinetic model. In addition, the result follows the pseudo second order because it has undergone chemisorption process which involves chemical adsorbate.



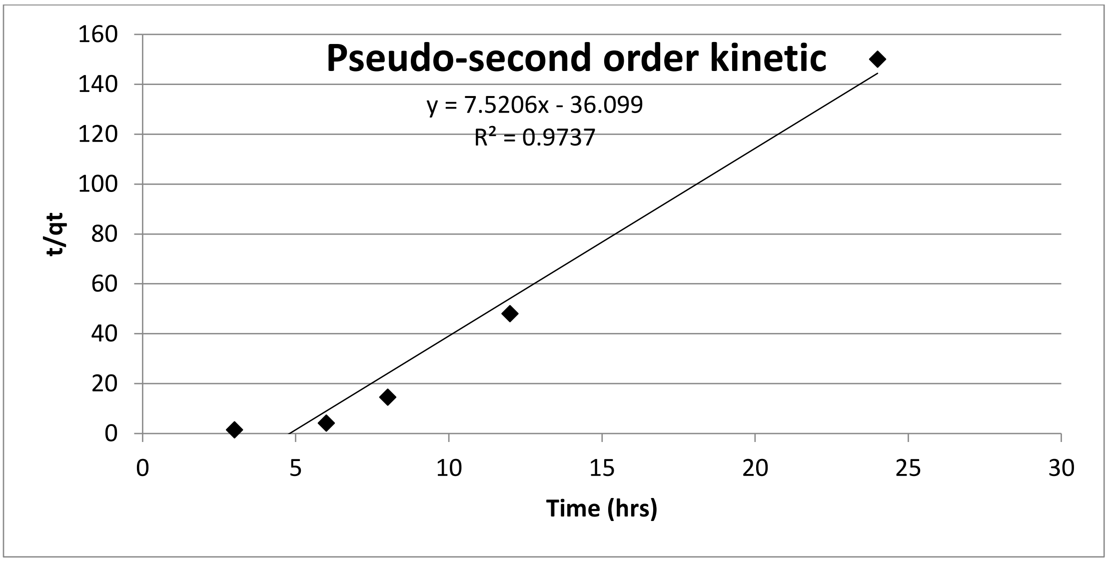
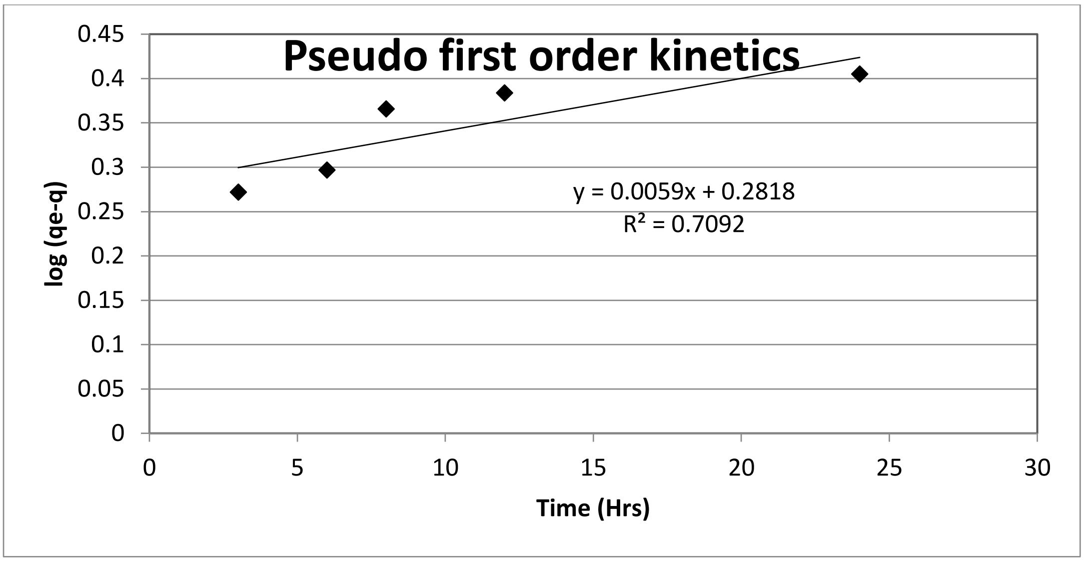


FIGURE 26: Pseudo-first & second order of **50 mg/L**

As can be seen from Figure 26 above, the data indicates that the pseudo-first order kinetic model has a lower value compared to pseudo-second order kinetic. Thus, the result from this experiment obeys the pseudo-second order kinetic model. In addition, the result follows the pseudo second order because it has undergone chemisorption process which the adsorption process involves chemical adsorbate.



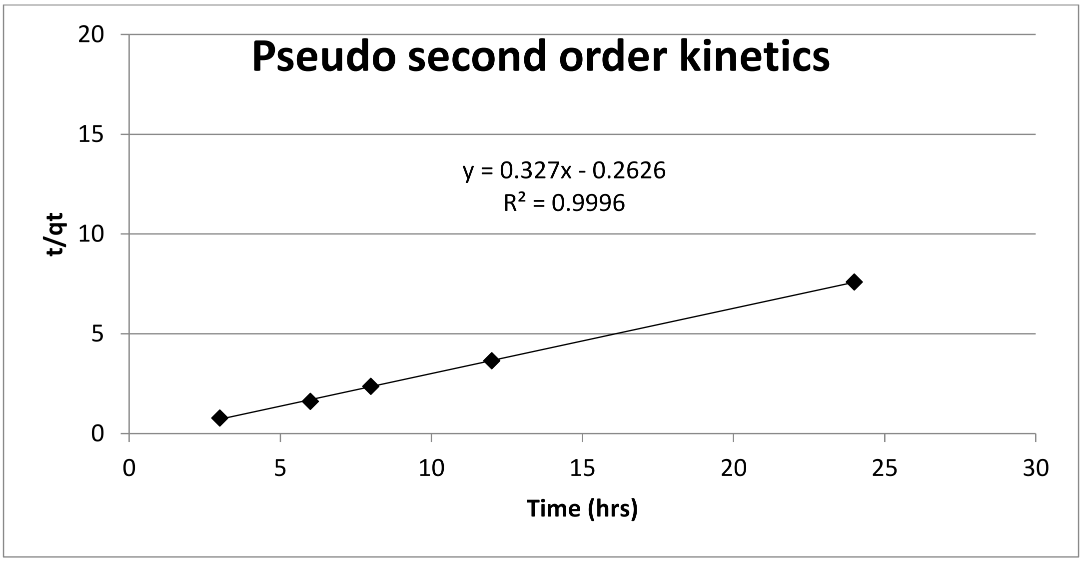


FIGURE 27: Pseudo-first & second order of **100 mg/L**

As can be seen from Figure 26 above, the data indicates that the pseudo-first order kinetic model has a lower value compared to pseudo-second order kinetic. Thus, the result from this experiment obeys the pseudo-second order kinetic model. In addition, the result follows the pseudo second order because it has undergone chemisorption process which involves chemical adsorbate.

4.4 Costing

In the industry, cost is the most important aspect that needs to be considered. The cost for the experiment should be cheaper than the alternatives that have already been used. The cost of the adsorbent is part of the decision making to allow researchers industrial people to opt for a more cost efficient and economical adsorption technique. Therefore, this experiment conducted ensures that the cost is low while still giving the most accurate result in accordance to the removal of heavy metal.

Aspects	Sludge Activated Carbon (RM)
Water consumption (approx. max 10)	3.00
Heating (Carbonization)	15.70
Chemicals	186.85
Overhead Cost (approx.)	100.00
Total / kg (RM)	**305.55**

TABLE 7: Cost of adsorbent per kilogram

Referring to table 7, Biomass based activated carbon is chosen for this experiment as it has a lower cost per kilogram compared to the cost of commercial activated carbon. The cost for the biomass based activated carbon is RM300 per kilogram which is produced from the sewage treatment plant. Biomass based activated carbon is not only eco-friendly. It is also efficient in removing heavy metals from the industrial wastewater.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The activation of the carbonization reduces the waste of sludge due to its high adsorption capacities from the available binding sites that occur in the adsorbate. The best heavy metal ion removal happens at the contact time of 24 hours and adsorbent dosage of all the concentration of zinc metal which are 10mg/L, 50mg/L and 100mg/L.

Referring to the results of the experiment, the best sample which achieves the highest removal efficiency is sample 10g/L of SAC dosage at a contact time of 24 hours at 100mg/L zinc concentration.

In conclusion, the results achieved follows the Freundlich isotherm model which indicates that monolayer occur at the surface of the adsorbate. The results are also is in line with pseudo-second order kinetic model compared to the pseudo-first order kinetic model and it also indicates that the experiment has undergone chemisorption process which the adsorption process involved with chemical adsorbate.

5.2 Recommendation

The recommendation that can be included from this experiment is to incorporate the use of this experiment with different types of wastes such as agricultural wastes from palm oil, coconut husk and rice husk. The different adsorption rate, activation time and temperature of the wastes can be altered accordingly. From this experiment, the amount of toxic waste and pollutions that are happening currently can be slowly reduced in the future by utilizing this experiment to remove heavy metals from different industrial wastes which indirectly reduces the amount of pollution and toxicity in the environment.

Another recommendation is referring to the timeline of this experiment. The timeline should be given a longer span to allow students to better research on the topic and experiments that are conducted in this FYP topic. If the timeline given is longer, the student is able to further experiment with not only a single parameter but also different parameters using different types of heavy metals which in return allows for a better study of this adsorption topic.

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APPENDICES

A. Activated Sludge

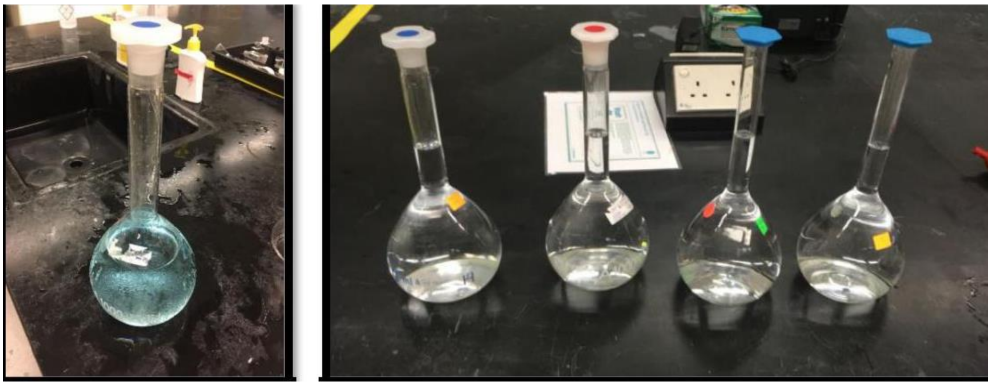
Sludge from UTP STP drying bed dried in an oven at 105°C for 24 hours until all moisture evaporated and a constant weight was achieved.

B. Incineration

About 30 g of the dried sludge was weighed, incinerated in a tube furnace at 500 °C for 3 hours. Heating rate of 10 °C/min, nitrogen flow rate of about one hundred millilitre (100 mL/min) was maintained during the production process.

C. Adsorbate Preparation

Preparation of sample and blank sample



Preparation of Stock Solution using equation of equilibrium

10 mg/L of zinc solution, 1:2 Dilution,

50 mg/L of zinc solution, 1:10 Dilution

100 mg/L zinc solution, 1:20 Dilution

The conical flasks were clamped in an orbital shaker and agitated for 3, 6, 8, 12 and 24 hours, respectively. The agitation speed was maintained at 150 rpm throughout the study