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Adsorption of Cadmium and Zinc onto *Stenochlaena palustris* (Paku Midin) : Potential agent for removal or recovery in aqueous solutions

By

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Mohd Akmal bin Hashim

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Adsorption of Cadmium (Cd) and Zinc (Zn) onto *Stenochlaena palustris* (Paku Midin) : Potential agent for removal or recovery in aqueous solutions.

Abstract

The ability of ***Stenochlaena palustris*** (Paku Midin) to remove Cadmium and Zinc from aqueous solution by adsorption was studied. The effects of Contact Time, pH and temperature of the solution, initial metal concentration prior to loading, presents of other metal ions, and adsorbent size/dose were studied in batch experiments . For this study, the weight and size of the adsorbent material were fixed. The weight of the adsorbent material used were set at 0.5 gram and size of #120 based on the use Screen sieves. The initial metal concentration are also been fixed at 1ppm and 2ppm for Zinc and Cadmium respectively. The results have shown that the leaves of Paku Midin can adsorp Cadmium up to 86.83 percent at 75 minutes contact time, while the stems can adsorp 83.36 percent at 30 minutes. After 30 and 75 minutes, the adsorption capacity of the adsorbent has shown reduction value, which indicate that 30 minutes and 75 minutes are the optimum contact time for Paku Midin stems and leaves respectively. However, the adsorbent was uneffective against Zinc adsorption or in other words the adsorbent cannot adsorp Zinc from the aqueous solution that have been prepared. For pH effect analysis, the leaves seems to show higher capacity of Cadmium adsorption at acidic or basic pH conditions, but less effective in near neutral pH condition. As for the stem of Paku Midin, adsorption of Cadmium seems to be better at pH below and above 5. For temperature study, the results has shown that both Paku Midin stems and leaves are more effective at higher temperature condition .

Keupayaan ***Stenochlaena palustris*** atau Paku Midin untuk membuang logam Kadmium dan Zink daripada larutan akueus secara perlekatan telah dikaji. Kesan-kesan seperti tempoh masa interaksi, pH dan suhu larutan, kepekatan awal logam sebelum kemasukan bahan pelekat, kehadiran logam asing dan saiz/dos bahan pelekat telah dikaji secara berperingkat dalam eksperimen ini. Dalam ujikaji ini, saiz dan berat bahan pelekat telah ditetapkan. Berat bahan pelekat yang digunakan telah ditetapkan pada 0.5 gram dan bersaiz #120 berpandukan kepada penggunaan penapis Screen. Kepekatan awal logam telah ditetapkan pada 1ppm dan 2ppm masing-masing untuk Zink dan Kadmium. Keputusan telah menunjukkan daun Paku Midin berupaya untuk melekat kepada logam Kadmium sehingga 86.83 peratus pada 75 minit tempoh interaksi, manakala batang Paku Midin pula berupaya melekat sehingga 83.36 peratus pada 30 minit tempoh interaksi. Selepas 30 dan 75 minit, didapati keupayaan pelekatan bahan pelekat telah menunjukkan nilai penurunan, di mana ini menandakan 30 minit dan 75 minit adalah tempoh interaksi optimum bagi masing-masing batang dan daun Paku Midin. Walaubagaimanapun, bahan pelekat ini didapati tidak efektif untuk pelekatan logam Zink atau dalam erti kata lain ia gagal untuk melekat kepada logam Zink di dalam larutan yang telah disediakan. Bagi analisa kesan pH, daun Paku Midin telah menunjukkan keupayaan pelekatan kepada logam Kadmium yang lebih baik pada keadaan pH berasid atau alkali, tetapi kurang efektif pada keadaan hampir ke pH neutral. Bagi batang Paku Midin pula, keupayaan pelekatan kepada logam Kadmium didapati berlaku lebih baik pada pH kurang dan lebih daripada 5. Seterusnya bagi analisa kesan suhu, keputusan telah menunjukkan bahawa kedua-dua batang dan daun Paku Midin adalah lebih efektif pada keadaan suhu yang lebih tinggi.

1. Introduction

1.1 Metals Pollution

Some agricultural activities, industrial and domestic waste pollute water bodies with heavy metals. These could in the end reach body tissues via the food chain. Beside pollution due to organic materials, the toxicity of heavy metals on aquatic organisms has been the subject of interest to biologist for many years The release of heavy metals into the environment by industrial activities is a serious environmental problem because they tend to remain indefinitely, circulating and eventually accumulating throughout the food chain (K.Periasamy and C. Namasivayam,1994). Adsorption of trace metals onto natural particulate matter can play an important role in determining trace metal speciation in many aquatic system (Laxen D.P.H , 1983).

Figure 1.1 : Industrial Pollution like the above can contribute to degradation of the environment .

(www.ithaca.edu/biology/278_Cadmiumarsenic.ppt.) Accessed on 12 Feb 2008.

Among the different types of pollution, industrial waste constitutes the major source of various kinds of metal pollution in natural water (R.M. Harrison and D.P.H.Laxen, 1983). The important toxic metals are **Cadmium (Cd), Zinc (Zn),** Chromium (Cr), Lead (Pb) and Copper (Cu) which finds their way to water bodies through wastewater from such industries as metal-plating industries of Cadmium, nickel batteries, pigment, stabilizers and also alloys (Low K.S and Lee C.K , 1991).

Different conventional processes (precipitation, ion exchange, electrochemical processes and/or membrane processes) are usually applied to the treatment of industrial effluents but the application of such processes is often limited because of technical or economic constraints (Veglio F. et al, 2003). Nowadays, different methods can be used for removing metals, including filtration, chemical precipitation, coagulation, solvent extraction, electrolysis, ion exchange, membrane process and **adsorption** (Patterson J.W , 1997). However, ion exchange and **adsorption** are the most common and effective processes for this purpose. Activated carbon and different types of ion-exchange resins are very often used in adsorption processes. However their high cost in production and regeneration have encouraged researchers to look for low-cost replenishable sorbing materials for the purpose of removal of heavy metals in an effort to reduce pollution as well as help in recycling of the indispensible metals.

Adsorption of heavy metals is one of the possible alternative technologies involved in the removal of toxic metals from industrial waste streams and natural waters using low-cost adsorbents. A low – cost sorbent is hereby defined as one which is abundant in nature, or is a by-product or waste material from another industry. Such materials could be an alternative for the conventional sorbents (Kratochvil D. & Volesky B. , 1998). Various waste materials produced as a result of different industrial processes, such as pine bark (Al-Asheh S. and Duvnjak Z. , 1998), cork and yohimbe bark (Villaescusa I. et al. ,2000), spent grain (Low K.S. et al., 2000), peanut hull pellets (Johnson P.D. et al. ,2002), and rice milling by-products (Teixeira C.R. & Zezzi,2004) have been studied for the decontamination of metal-containing effluents.

Adsorption process is a potential and interesting alternative to conventional processes for the removal of metals, such as ion exchange processes (Pagnanelli F. et al ,2003). It is a rapid, reversible, economical and ecofriendly technology in contrast to conventional chemical methods of removing metal ions from industrial effluents. Adsorption of metal ions from aquatic system using fruit wastes (eg. fruit peel orange), herbs, peanut skin, barley straw, pine bark, microbial biomass, including algae, fungi and bacteria has gained importance in recent years (Gadd G.M. & White C. ,1993). The sorption of metals by this kind of material might be due to the presence of carboxyl, hydroxyl, sulphate, phosphate, and amino groups that can bind metal ions. As a result, the adsorbent materials are very usefull for the cleaning of used solution or system contaminated by the metals or other pollutants. For instance, in industrial area where contaminans such as detergents and metals are usually released to the environment, the natural adsorbent material such as the Paku Midin can be used to treat the polluted river or streams.

1.2 The Adsorbent Material (*Stenochlaena Palustris*)

In this study, the researcher have tried to investigate the potential of some wild plant from the environment as an alternative low-cost metal sorbents. The wild plant that the researcher have choosed were known as *Stenochlaena palustris* or also called as **'Paku Midin'** by the locals. This type of wild plant can be easily found near the wet ground, scrambling in open places and climbing in secondary forest. Sometimes it also can be found in lowlands and hills (Idris M. Said, 2005). In the present work, this sorbent was investigated for Cadmium and Zinc removal from aqueous solution. Both metals were released into the environment from different anthropogenic sources e.g. metal processing industries, battery and paint manufacturing, fossil fuel combustion, etc. There, they tend to accumulate and become concentrated throughout the food chain. This aspect, coupled with their persistence, results in a serious health hazard threatening water supplies and populations (Environmental Protection Agency, U.S, 1999).

The brown green coloured Paku Midin was bought from Pasar Besar Siti Khadijah, Kota Bharu. It was then washed with tap water several times to remove the impurities. The leaves of Paku Midin was separated from their stems. The separated stems and leaves were then placed in separate aluminium foil. Both stems and leaves were completely dried inside an oven for four days at a temperature of 60°C. The dried leaves and stems were then cut into small pieces and semi-pulverised using a domestic blender. After that, the semi-pulverised materials were sieved by using a screen sieve of #120 mesh size. Lastly, we shall have the processed samples of the adsorbent materials

TAXONOMY & INFORMATION OF 'PAKU MIDIN'

Kingdom	PLANTAE
Class	Pteridopsida
Order	Athyriales
Family	Blechnaceae
Genus	Stenochlaena
Species	Stenochlaena palustris
Vernacular name	Paku Midin
Major Area	Sabah (Malaysia), Peninsular Malaysia
Distribution	Ranau
Habitat	Common in rather wet ground, scrambling in open places and climbing in secondary forest, in the lowlands and hills
Global distribution	Australia, India, Japan, Singapore
Character	The fronds are pinnate, The broad sterile pinnae with sharply toothed margins. The lamina of fertile pinnae is reduced and the lower surface covered with sporangia. Young fronds are red
Uses	The red young fronds are edible and are used as vegetable. The juice is used to cure fevers and the long rhizomes to bind baskets and fish traps

Table 1.1 : Taxonomy and Information of *Stenochlaena palustris*

1.3 Cadmium and Zinc (Heavy Metals)

Heavy metals exist in aqueous waste streams of many industries like metallurgical/metal manufacturing and electroplating, chemical manufacturing, printing, dye and paint industry, paper, textile, refinery and petrochemicals, leather goods manufacturing, fertilizer and pesticides industry (Srivastava V.C. et al, 2006). Heavy metals have cumulative effects and tend to accumulate in the living organisms and could cause various diseases and disorders.

According to Klaassen et al (2001), Cadmium was relatively a new metal in terms of humans toxicity. There were many sources of Cadmium such as natural rock weathering, copper, lead and zinc smelting, auto exhaust and also cigarette smoke (a cigarette contains 1-2 ug Cd). Cadmium were mainly used for metal plating , making nickel-cadmium batteries, solders, paint pigments (blue), plastic stabilizer, photographic chemicals, and also fungicides.

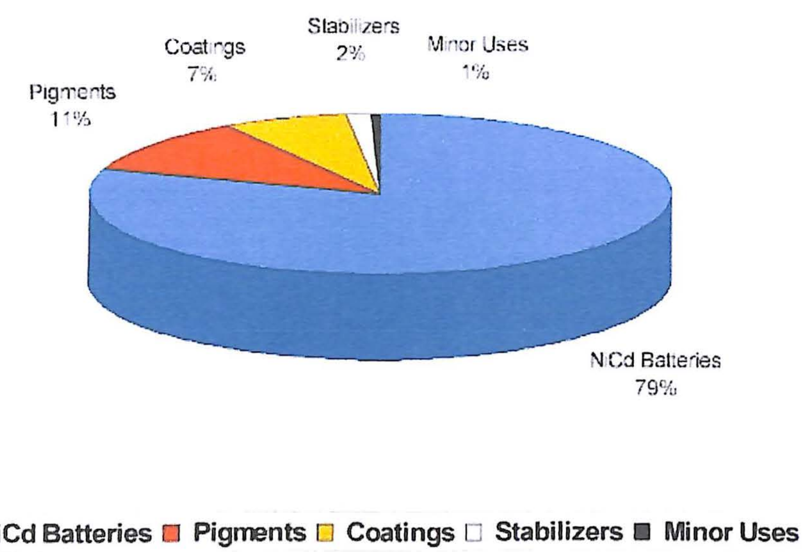


Figure 1.5 : Cadmium uses
(www.ithaca.edu/biology/278_Cadmiumarsenic.ppt.) Accessed on 12 Feb 2008

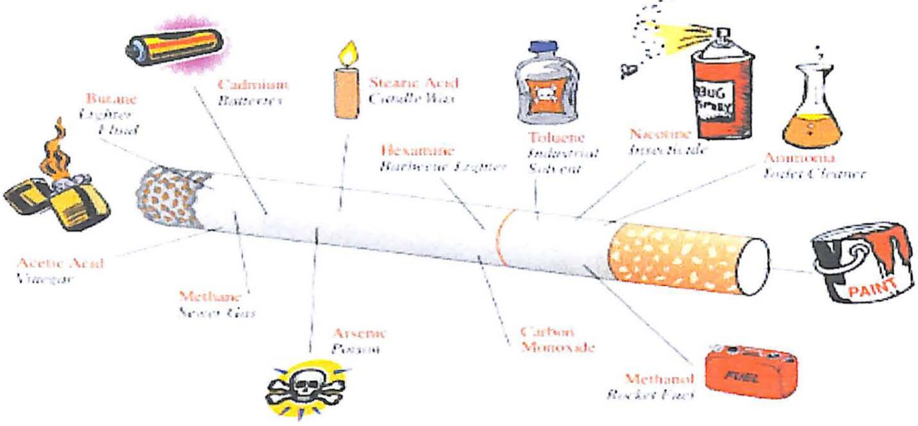


Figure 1.6 : Cadmium inside a cigarette
(www.ithaca.edu/biology/278_Cadmiumarsenic.ppt.) Accessed on 12 Feb 2008

Futhermore, Cadmium [Cd] is a non-essential and non-biodegradable metals which slowly accumulates in the human body, usually from food-chain. The body slowly releases adsorbed Cadmium(Cd) which has a biological half-life of more than 10 years. As a consequence, the Cadmium(Cd) content of the kidney increases throughout life. Ingested Cadmium is transported to other organs by the blood, where it is bound by glutathione and haemoglobin in the red blood cells (Rabenstein D.L. et al, 1983). It is found that 50-75% of total body Cd is found in liver and kidney (Klaassen et al., 2001). Consumption of rice containing high concentrations of cadmium led to a surge in the **'Itai-Itai'** disease in Japan in 1955 (Friberg L. et al, 1979).

Figure 1.7: **Itai-itai** victim

(www.ithaca.edu/biology/278_Cadmiumarsenic.ppt.) Accessed on 12 Feb 2008

In humans, Zinc [Zn(II)] are found in over 20 metalloenzymes, including several that are involved in nucleic acid metabolism. Excess ingestion of Zn(II) may result in acute gastrointestinal disturbances accompanied with nausea. Instances of acute toxicity have occurred from ingestion of fruit juices that were stored in galvanized (zinc plated) steel containers (S.P. Mishra et al, 1997). Due to toxicity of metals, the Ministry of Environment and Forests, Government of India has set Minimal National Standards of 0.2 and 5.0 mg/l, respectively, for Cd(II) and Zn(II) for safe discharge of the effluents containing these metal ions into surface waters (MINAS, Pollution control acts, Govt. of India, New Delhi, 2001).

Much of the work on the adsorption of heavy metal ions by various kinds of adsorbents has focused on the uptake of single metal ions. As a result, no information is available in literature for the simultaneous removal of Cd(II) and Zn(II) ions by 'Paku Midin' or even other natural sorbents, for that matter.

2. Objective of the study

The present study aims to examine the feasibility of using ‘Paku Midin’ as an adsorbent for the removal of Cd(II) and Zn(II) metal ions from aqueous solutions. The effect of pH, influence of contact time and also temperature (T/°C) on metal adsorption, were also studied.

3. Materials and methods

3.1 Preparation of adsorbent (Paku Midin)

The brown-green coloured *Stenochlaena palustris* sp used in the present study were bought from Pasar Besar Siti Khadijah, Kota Bharu, Kelantan, Malaysia . The plant were then washed with water several times to remove impurities. The washing process was continued till the plant were clean and the wash water contains no dirt. The washed plant were then separated according to their stem and leaves. Next, the separated stem and leaves were placed in the aluminium foil separately. Both (stem and leaves) were completely dried inside an oven for 4 days under the temperature of 60°C. The resulting product was directly used as adsorbent without any pretreatment. The dried plant were then cut into small pieces and were pulverised using domestic blender. In this study the pulverised materials were sieved by using the screen sieves. The adsorbent materials that been used in this study were sieve under the size of #120 mesh size. Both (stem and leaves) were then directly used as adsorbents without any pretreatment.

3.2 Preparation of Chemicals and Metal solutions

3.2.1 Preparation of Stock Solutions

3.2.1.1 Cadmium

Stock solution of Cadmium concentration 1000 mg/L was prepared by dissolving 3.93 g of 100% $Cd{\cdot}H_2O$ (Merck) in 1000 mL of distilled water. The solution was prepared using standard flasks. The range of concentration of the prepared metal solutions varied between 20 and 100 mg/L and they were prepared by diluting the cadmium stock solution, which were obtained by dissolving in deionized water.

3.2..1.2 Zinc

Stock solution of Zinc concentration 500 mg/L was prepared by dissolving 0.5 g of Zinc metal in a minimum volume of (1+1) HCL and dilute to 1 Litre with 1% (v/v) HCL.

3.2.2 Preparation of Standard Solution for Calibration Curve

3.2.2.1 Standard Solution of Cadmium.

Before running the analysis on the desired elements, we have to prepare the calibration sample or the standard solution in order to obtain the **calibration curve** . It is vital since to make sure the analysis that we try to run will bring out valid result and also to ensure the machine (AAS) or stock solution of chemicals that been used were stable and good in condition. In this study, we used the principle of **serial dilution** in order to obtain three different concentration of standard solution. For Cadmium, the first step is to prepare 100ml of **2ppm@2mg/L** standard solutions of it. The reason why we choosed this concentration was because it is the highest level of Cadmium concentration that can be detected through the AAS analyzer machine.

Next, will be the methods that we used in preparing the **standard solution of Cadmium** in order to obtain the calibration curve . Firstly, 200µl of stock solution of Cadmium were pipetted onto 100ml volumetric flask. Then we add deionized water to the stock solution until it reach 100ml. Next, from the solution that have been prepared, 50ml from it, were then pipetted into another 100ml clean volumetric flask that have been rinsed with deionized water . After that, once again we add deionized water to the 50ml solution until it reach 100ml.

Next, as for the third concentration, we repeat the same method as before. 50ml of the solution from the second concentration were pipetted into another 100ml clean volumetric flask that also have been rinsed with deionized water before. Then again, we topup the solution with deionized water until it reach 100ml. After finished, we will have three different concentrations of standard solutions of Cadmium which are 2ppm,1ppm and 0.5ppm respectively.

3.2.2.2 Standard Solution of Zinc.

Same as preparing the standard solution for Cadmium, we used the principle of serial dilution in order to obtain three different concentration of standard solution. As for Zinc, the first step was to prepare the 100ml of 1ppm@1mg/L standard solutions of it. The reason why we choosed this concentration was because it is the highest level of Zinc concentration that can be detected through the AAS analyzer machine.

Firstly, 100µl of stock solution of Cadmium were pipetted onto 100ml volumetric flask. Then the deionized water was added to the stock solution until it reach 100ml. Next, from the solution that have been prepared, 50ml from it, were then pipetted into another 100ml clean volumetric flask that have been rinsed with deionized water . After that, once again the deionized water was added to the 50ml solution until it reach 100ml.

Next, as for the third concentration, the same method was repeated. 50ml of the solution from the second concentration were pipetted into another 100ml clean volumetric flask that also have been rinsed with deionized water before. Then again, we topup the solution with deionized water until it reach 100ml. After finished, the researcher will have three different concentrations of standard solutions of Zinc which are 1ppm, 0.5ppm and 0.25ppm respectively.

3.2.3 Preparation of Standard Solution from the Stock solution.

In this study, we will analysed the ability of natural adsorbent to adsorp certain amount of metals that have been loaded in an aqueous solution. So, we need to prepare the standard solution where it will contains certain amount of metals (Cadmium or Zinc) . As for Cadmium we need to prepare 2ppm or 2mg/L of standard solutions from the stock solution which is 1000ppm in concentration. In order to prepare the standard solution of 2ppm Cadmium, firstly we need to pipet 200µl of stock solution of Cadmium onto 100ml clean volumetric flask that have been rinsed with deionised water. Next, the stock solution were then being topup with deionized water until it reach 100ml. As for Zinc, we need to prepare 1ppm or 1mg/L of standard solutions from the stock solution which is also 1000ppm in concentration. Firstly, 100µl stock solution of Zinc were pipetted onto 100ml clean volumetric flask that have been rinsed with deionised water. Then the stock solution then being topup with deionized water until it reach 100m

3.2.4 Preparation of Standard Solution with different pH.

In this study, we also have investigate the effect of different pH conditions against the adsoption of Cadmium by the adsorbent. We did not perform the same study on Zinc adsorption since the results have shown that the adsorbent was uneffective or in other words the adsorbent cannot adsorp Zinc from the aqueous solution that have been prepared.

In purpose to study the effect of different pH conditions against the adsorption of Cadmium by the adsorbent, we need to prepare the standard solution of Cadmium with different pH. Hydrochloric acid (HCL) and Sodium Hydroxide(NaOH) were used in order to adjust the pH of the standard solution. pH meter was also been used for the purpose to determine the pH of the solution that have been adjusted. Three type of samples need to be prepared for each pH been studied in case to avoid interference or noise effect. The three type of samples were Blank sample, Calibration sample and also the Adsorbent to be study sample.

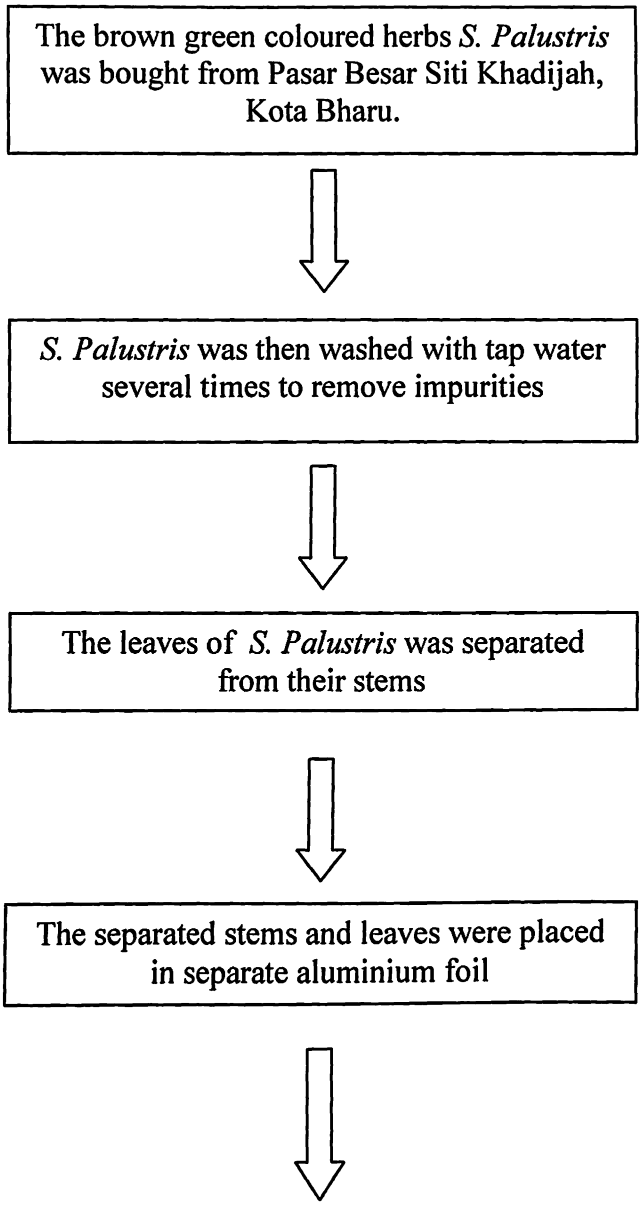
3.2.5 Preparation of Standard Solution at different temperature.

Furthermore, we also have investigate the effect of different temperature (T/°C) conditions against the adsoption of Cadmium by the adsorbent. Same as stated before, we did not perform the same study on Zinc adsorption since the results have shown that the adsorbent was uneffective or in other words the adsorbent cannot adsorp Zinc from the aqueous solution that have been prepared.

In order to study the effect of different temperature (T/°C) conditions against the adsorption of Cadmium by the adsorbent, we firstly need to prepare the standard solution of Cadmium. From the standard solution, the control and the study sample been prepared. Adsorbent were added to the standard solution in order to make the study sample. After that both of the control and the study sample need to be left inside a waterbath at different temperature (T/°C). The temperature that have been choosed to be study were 20°C, 30 °C, 40 °C, 50 °C and 60 °C. Thermometer was also been used for the purpose to check the temperature (T/°C) inside the waterbath that have been adjusted. The leaves samples need to be left for 75 minutes while the stem sample only for 30 minutes. Both of the duration, 75 and 30 minutes have been chosen because it was the optimum contact time for the adsorption of Cadmium for each samples respectively.

3.3. Flowchart of The Research Methodology

3.3.1 Preparation of the adsorbent materials



Both stems and leaves were completely dried inside an oven for four days at a temperature of 60°C

↓

The dried leaves and stems were cut into small pieces and semi-pulverised using a domestic blender

↓

The semi-pulverised materials were sieved by using a screen sieve of #120 mesh size

↓

PROCESSED SAMPLES / ADSORBENT MATERIALS

3.3.2 Methodology to study the effect of **Contact Time**

PROCESSED SAMPLES / ADSORBENT MATERIALS

↓

Weigh 0.5 gram of the sample using Sartorius Electronic Balance

↓

Pour the adsorbent materials into clean universal bottle

↓

Pipette 10ml of standard solution containing 2ppm Cadmium that have been prepared into the universal bottle

↓

Pipette another 10ml of standard solution containing 2ppm Cadmium into another clean universal bottle without the adsorbent. This will be used as the negative control in this study

↓

The universal bottles were left on rotator for different time intervals ranging from 15- 75 minutes

↓

Filtrate the sample into another clean universal bottle using the fiter paper and filter funnel

↓

Measure the concentration of elements (Cd/Zn) that was still remaining in the sample by using the A.A.S machine

3.3.3 Methodology to study the effect of **pH**

3.3.3.1 Preparation of **Blank Sample**

500ml of Deionized water was pour into a clean beaker

↓

The pH of the deionized water was adjusted with 0.1M NaOH and 0.1M HCl

↓

Different pH of deionized water (pH3, 5, 7, and 9) / BLANK SAMPLE

3.3.3.2 Preparation of **Calibration Solution**

200ul stock solution of Cadmium was pipette into a clean 100ml volumetric flask

↓

Then, the stock solution was top up with deionized water with spesific pH (3, 5, 7 or 9) until 100ml

↓

2ppm of standard solution been prepared

↓

Perform serial dilution.
50ml of 2ppm standard solution was pipette into another clean 100ml volumetric flask.

↓

Top up solution with deionized water to 100ml

↓

1ppm of standard solution been prepared

↓

50ml of 1ppm standard solution was again pipette into another clean 100ml volumetric flask.

↓

Top up solution with deionized water until 100ml

↓

0.5ppm of standard solution been prepared

3.3.3.3 Preparation of **Samples**

Standard solution of 2ppm Cadmium with different pH

↓

10ml was pipette into a clean universal bottle containing 0.5 gram of adsorbent materials

↓

Samples were left on rotator. The duration depends on the part of the adsorbent material :

- Stems – 30 minutes
- Leaves – 75 minutes

↓

Filtrate the sample into another clean universal bottle using the fiter paper and filter funnel

⇩

Measure the concentration of elements (Cd) that was still remaining in the sample by using the A.A.S machine

3.3.4 Methodology to study the effect of **Temperature**

PROCESSED SAMPLES / ADSORBENT MATERIALS

⇩

Weigh 0.5 gram of the sample using Sartorius Electronic Balance

⇩

Pour the adsorbent materials into clean universal bottle

⇩

Pipette 10ml of standard solution containing 2ppm Cadmium that have been prepared into the universal bottle

⇩

Pipette another 10ml of standard solution containing 2ppm Cadmium into another clean universal bottle without the adsorbent. This will be used as the negative control in this study

↓

Left the samples inside a waterbath for several minutes according to their parts ; 30 minutes for stems and 75 minutes for leaves. The waterbath was set to different temperature ranging from 30°C to 60°C. As for 20°C, the sample was left in air-conditioned room

↓

Filtrate the sample into another clean universal bottle using the fiter paper and filter funnel

↓

Measure the concentration of elements (Cd) that was still remaining in the sample by using the A.A.S machine