Faculty of Resource Science and Technology

Activated Carbon from Pepper Waste for Heavy Metals Adsorption

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List of Abbreviations

AC: Activated carbon

CAC: Commercial activated carbon

SEM: Scanning electron microscope

FAAS : Flame atomic absorption spectroscopy

US EPA : United State Environment Protection Agency

MPB: Malaysian Pepper Board

Pb : Lead

Zn : Zinc

Cd : Cadmium

Cu : Copper

Hg : Mercury

As : Arsenic

Al : Aluminum

Zn : Zinc

Fe : Iron

Co : Cobalt

Mn : Mangenese

Ni : Nickel

mL: mililiter

nm: nanometer

ppm : part per million

g : gram

$gmol^{-1}$: gram per mol

mg/kg : milligram per kilogram

°C: degree Celcius

rpm: Rotary per minute

C_i : the initial metal solution concentration (ppm)

C_f : the final metal solution concentration (ppm)

H_2SO_4: Sulfuric acid

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Activated carbon from pepper waste for heavy metals adsorption

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ABSTRACT

In this study, the ability of activated carbon (AC) from *Piper nigrum* waste to remove lead (Pb), iron (Fe) and zinc (Zn) from aqueous solution was investigated. The AC was prepared by using chemical activation process. The Scanning Electron Microscope (SEM) images showed that the carbon before activation has less pores compared to carbon after activation. The performance of AC was studied by varying the adsorbent dosage, initial metals concentration, and agitation time on the metals removal. The adsorption ability of pepper waste based AC was compared with adsorption ability of commercial activated carbon (CAC). Pepper waste based AC showed a favorable result where it was able to adsorb Pb and Fe metals up to 89.9% and 46.0%, respectively. Zn metals adsorption does not show a good result where it only adsorbed 31.4%. The study also showed that the removal efficiency of the pepper waste based AC was greatly affected by the adsorbent dosage and initial metal concentrations while contact time showed less dependency. The use of pepper waste as AC can help to overcome the abundance of pepper waste and can be used as a low cost material for water treatment.

Keywords: *Piper nigrum* waste, activated carbon, heavy metals, adsorption.

ABSTRAK

Dalam kajian ini, kemampuan karbon teraktif dari sisa P. nigrum untuk mengeluarkan logam plumbum (Pb), ferum (Fe) dan zink (Zn) daripada larutan akueus telah dikaji. Karbon teraktif ini telah disediakan dengan menggunakan proses pengaktifan kimia. Imej Mikroskop Pengimbasan Elektron (SEM) menunjukkan bahawa karbon sebelum diaktifkan mempunyai kurang pori berbanding karbon yang telah diaktifkan. Kebolehan karbon teraktif ini telah dikaji dengan mempelbagaikan dos adsorben, kepekatan awal logam, dan masa agitasi bagi pengeluaran logam-logam. Kemampuan menyerap karbon teraktif dari sisa lada telah dibandingkan dengan kemampuan menyerap karbon teraktif komersial. Karbon teraktif dari sisa lada menunjukkan keputusan yang memberansangkan kerana ia mampu menyerap logam Pb dan Fe sehingga 89.9% dan 46.0%, masing-masing. Penyerapan logam Zn tidak menunjukkan keputusan yang baik kerana ia hanya menyerap 31.3% sahaja. Kajian ini turut menunjukkan bahawa kecekapan penyingkiran karbon teraktif dari sisa lada adalah sangat dipengaruhi oleh dos adsorben dan kepekatan awal logam manakala masa agitasi menunjukkan kurang pengaruh. Penggunaan sisa lada sebagai karbon teraktif dapat mengatasi masalah akumulasi sisa lada dan dapat digunakan sebagai bahan inisiatif kos kendah bagi merawat air.

Kata kunci: sisa Piper nigrum, karbon teraktif, logam berat, penyerapan.

1. Introduction

1.1. General introduction

Spice is a dried ingredient that has aromatic or pungent smell and it has been used in flavoring the dishes. One of the most commonly used spices is black pepper. Black pepper is a flowering vine plant which comes from the family of Piperaceae. It comes with a scientific name called *Piper nigrum.* Malaysian Pepper Board that has been established under the Ministry of Plantation Industries and Commodities of Malaysia are responsible in handling and managing all the activities in pepper's production in Malaysia. According to Desai (2014), wise production of black and white pepper in Malaysia has increased from 19000 tonnes in 2012 to 19500 tonnes in 2013 and 21000 tonnes is estimated to be the production in 2014. However, the increase of the production of pepper has caused the increase in waste production such as pepper stalks, pinhead, light berries and pepper dusk which are all produced from the dry and wet cleaning process of the pepper. Wastes produced from pepper production need to be converted into the usable form such as activated carbon in order to avoid continuous accumulation of such waste in the environment.

All AC can be made from organic parent sources with distinct carbonization and manufacturing process. There are various types of agriculture biomass or waste such as peanut hull, wood sawdust, rice husk, wheat straws, banana pitch, and orange peel that has been utilized for the production of AC. AC is a form of carbon that need to undergo activation process to make it riddled with small, low volume pores that can raise the surface area that suitable for absorption or chemical reactions. The high porosity and surface functional groups of the AC make it a good absorbent, catalysts supports and electrode materials (Martins & Fernandes, 2012).

There are two types of activation process namely physical activation and chemical activation (Manocha, 2003). Physical activation is a process that involves thermal process while chemical activation is a process that involves chemical such as acid, base, or salt to react with the carbon. Martins and Fernandes (2012) have found that AC that has high surface area can be produced from sodium salt of carboxyl methyl cellulose by low temperature activation that required quenching of the red-hot-carbonised material in hot water. The high porosity characteristics of an AC increase its surface area which makes it a better adsorbent.

1.2. Problem statement

P. nigrum production has increased year by year. Every pepper production can produce an average of 10 to 18 tonnes of waste per year. Production of AC from agriculture such as peanut hull, wood sawdust, rice husk, wheat straws, banana pitch, and orange peel have been identified as one possible way in converting waste into usable product. As such, pepper waste was converted to AC and evaluated as heavy metals adsorbent. Heavy metals such as lead (Pb), zinc (Zn), and iron (Fe) have contributed to water contamination. The conversion of pepper waste into AC may also overcome accumulation of unused waste disposal.

1.3. Objectives

The objectives of this study are to:-

i. prepare and characterize AC from pepper waste.

ii. evaluate the efficiency of AC from pepper waste for heavy metals (Pb, Zn, and Fe) adsorption.

iii. characterize the AC for surface morphology using SEM.

iv. compare the efficiency of the pepper waste based AC as heavy metals (Pb, Zn, and Fe) adsorbent with that of CAC.

2. Literature Review

2.1. Pepper waste

Pepper waste is all of the other part of pepper tree that is not involve in the pepper production for commercialization. All the by-products that have been obtained from the dry and wet cleaning processes of the pepper such as pepper stalks, pinhead, light berries and pepper dusk can be considered as waste. Accumulation of biomass in the environment can cause various environment issues if no proper measures are taken to overcome it.

2.2. Activated carbon

Activated carbon is a carbonaceous material that has porosity and surface functional group (Serp & Figueiredo, 2009). The porosity of the AC makes it such a good adsorbents, catalysts, catalysts supports and electrode materials while the surface functional groups can improve the chemical adsorption. AC are vastly used as adsorbent in technologies related to pollution abatement due to its porous characteristic and large adsorption capacity (Budinova, Petrov, Parra, & Baloutzov, 2008). According to Bansal and Goyal (2010), AC in its broadest sense is a term that comprises a large range of unorganised carbonaceous materials that can present with high porosity and a large surface area. The raw materials could comprise of cellulose residues, sawdust, coconuts shells, peat, coal, lignite, palm oil, petroleum coke, sago waste, pepper waste, and wood waste can be carbonized and activated at high temperature. The carbonaceous substance is treated with chemical activating agent such as sulphuric acid or zinc chloride. The mixture is then carbonized at a high temperature and it is followed by washing process with distilled water to remove the chemical activating agent. AC is a special and versatile adsorbent. They are used widely for the removal of unwanted odour, taste, colour, and other inorganic and organic impurities from industrial and domestic waste water, air purification in inhabited places, solvent recovery, food processing, restaurants, and chemical industries; in getting rid of colour from various syrups and pharmaceutical products; in air contamination control from industrial and automobile exhausts; in the purification of many chemicals, pharmaceutical and food products; and in a variety of gas-phase applications (Bansal & Goyal, 2010).

AC can be characterised based on their porosity since the AC has different pore size. The AC is viewed under the Scanning Electron Microscope (SEM) to be characterised. SEM is used to analyse the surface structure or morphology of the AC. According to Marsh and food processing Rodríguez-Reinoso (2006), the pore size of AC determine how the adsorption takes place in the narrow and wide pore as microporosity<2.0 nm, mesoporosity 2.0-50 nm and macroporosity>50 nm.

In order to activate the carbon, it needs to undergo activation process. There are two types of activation which is chemical activation and physical activation. Marsh and Rodríguez-Reinoso (2006) stated that chemical activation process involves co-carbonization process with zinc chloride, phosphoric acid or potassium hydroxide. This salt, acid and base gives different activation mechanisms. Zinc chloride promote water molecules extraction from lignocellulosic structures of parent materials, phosphoric acid chemically combine within the lignocellulosic structures and potassium hydroxide activates an existing carbon involving disintegration of structure following intercalation and some gasification by the hydroxide oxygen which makes it the most complex mechanisms (Marsh & Rodríguez-Reinoso, 2006). Physical activation also known as thermal activation of AC involves process of removing carbon within the pores by gasification with carbon dioxide or steam at a very high temperature. This process produces carbons that have different porosity.

2.3. Heavy Metals

Heavy metals are the metal elements that are essential to most of the living organisms at low concentration but it can be toxic at elevated concentration. Heavy metals can be found naturally in the environment since most of the heavy metals are the element of the earth itself but in a very small quantity. Human activities such as industrial activities, mining, and agriculture activities usually contribute to the increase of the heavy metals concentration in the environment. Heavy metals are composed of important elements such as iron (Fe), cobalt (Co), copper (Cu), and zinc (Zn) and some other toxic element that was known as mercury (Hg), cadmium (Cd), and lead (Pb). On the other hand, compounds containing Cr, Cu, Hg, Cd, Ni, Zn, and Pb were produced by industrial and metallic derivatives containing As, Cu, Sb, Co, Zn, Au, Cd, Pb, Cl, and C were also used in domestic activities (Ferguson & Kim, 1991; Abdulla & Chmielnicka, 1990). All of them cannot be degraded or destroyed in environments which mean that they are part of conservative pollutants (Kim et al., 2010).

2.3.1. Lead (Pb)

Lead (Pb) is a very soft, highly permeable, ductile and a relatively poor conductor of electricity with a bluish-white lustrous colour metal. Rodgers (2002) stated that the symbol Pb that represents lead is derived from the Latin word, *plumbum* since lead is the metals that have been used in the earlier day in making pipes for plumbing. It has atomic number of 82 and atomic mass of 207.2 $gmol^{-1}$. Pb is not an easily found metal. Usually Pb that has been found in the ore comes with copper, silver and zinc. It needs to be extracted in order to have a pure

Pb. Pb can be found naturally in the environment but most of the Pb that has been found in the environment results from human activities such as solid waste combustion, industrial processes and fuel combustion. It can accumulate in the soil or water bodies through rain flow or the travel of the small particle in the air. The present of Pb in the soil can disturb the function of the soil. The soil can be poisoned by the Pb. Pb in the environment is harmful to human and ecosystem due to its insusceptibility to degradation and high toxicity (Sekar, Sakthi, & Rengaraj, 2004). Pb that accumulates in the water bodies can affect the health of shellfish and disturb the function of the phytoplankton as an important source of oxygen production in seas. Level of Pb's elevation was detected in agricultural soils in urban area sides mainly due to waste discharge, gasoline burning, and so on (Machida, Kikuchi, Aikawa, & Tatsumoto, 2004).

2.3.2. Zinc (Zn)

Zinc (Zn) is another metal that found naturally in the environment. The origin of its name is obscure but may plausibly be thought to be derived from a German word which is *Zinke* that means spike or tooth according to the appearance of the metal itself (Greenwood & Earnshaw, 2001). It comes with atomic number of 30 and 65.37 $gmol^{-1}$ atomic mass. Zn can be found in some chemical forms such as hydrated ions, metal-inorganic complexes, or metal-organic complexes in natural water. Hydrated Zn cations can be hydrolysed to form Zn hydroxide (U.S. EPA, 1979). Although the Zn occurs naturally in soil, water and air but the rise of its concentration occur unnaturally. It can be caused by most of human activities such as industrial activity, mining, coal and waste combustion and steel processing. All of these activities make the soils become heavily contaminated with Zn especially at the Zn mining and refining area and at the area where the sewage sludge from industrial area are used as fertilizer. Besides that, Zn can be taken up by vegetables and plants which the normal range of Zn content is in the range of 15 to 100 mg/kg (Merian & Thomas, 1991). The increase of Zn concentration in the water and soil can affect the food chain since some aquatic life can accumulate Zn in their bodies while plant species can also be affected by the soil that rich with Zn. Microorganisms and earthworm activities in the soil can be affected by the Zn and this will finally cause the breakdown of organic matter become seriously slow down.

2.3.3. Iron (Fe)

Iron (Fe) is a metal that have 55.85 $gmol^{-1}$ of atomic mass and 26 in atomic number. Fe is lustrous and silvery in colour and it is fairly soft and readily worked when it is pure (Greenwood & Earnshaw, 2001). Fe is believe to be the 10th most abundant element in the universe since it is the most abundant element making up the earth by 34.6%. Fe is fundamental to most of living things, from microorganisms to human being. Fe also is an essential part of the haemoglobin. It helps the blood to transport oxygen throughout the body. Unfortunately, some Fe compounds such as Iron (III)-O-arsenite and Iron (III)-O-pentahydrate may be hazardous to the environment. These iron containing chemicals can enter into the environment through the uncontrolled release of industrial waste. Accumulation of Fe containing chemical can endanger most of living things.

2.4. Studies on Heavy Metals Adsorption by Activated Carbon

There are a numerous studies that have been done on AC based on different agriculture waste in order to produce the cost-effective heavy metal adsorbent. Sago waste, coconut shells, cotton stalks, dated pits, sugarcane, pumpkin stem, apple waste, and rice husk was part of the agriculture waste that has been studied in this case. Bernard, Jimoh, and Odigure (2013) have done a research on heavy metals removal from industrial wastewater by AC prepared from coconut shell. Based on their research, it is showed that coconut shells based AC can be efficiently used for removal of metals ions. Besides that, Kobya, Demirbas, Senturk, and Ince (2005) have done a study on adsorption of heavy metal ions from aqueous solutions by AC prepared from apricot stone. In this study, it is found that adsorption of metals are dependent on solution pH as the highest adsorption of the different metals occurred at different pH. Kadirvelu and Namasivayam (2002) have reported that carbon prepared from coirpith is expected to be an economical for metal ion adsorption from wastewater in their research on AC from coconut coirpith as Cd(II) adsorbent from aqueous solution.

3. Methodology

3.1. Samples collection and preparation

The sample of pepper waste was collected from Malaysian Pepper Board (MPB), Sarawak factory. The pepper wastes was carbonated and activated by the MPB. The samples were obtained in a form of fine powder.

3.2. Characterization of activated carbon

JEOL Scanning Electron Microscope (SEM) (Model JSM-6390LA) was used to analyse the surface structure or morphology of the pepper waste and AC before and after the activation. CAC is also analysed with this analysis. Besides, the pepper waste based AC and CAC after adsorption process was also analysed. The pepper waste based AC and CAC was dried in the oven at 30 °C overnight after adsorption and it was analysed under SEM.

3.3. Absorption study

3.3.1. Effect of adsorbent dosage

Different dosages of adsorbent ware tested. Adsorbent dosage was varied from 0.2 – 1.0 g. A blank control was prepared for every batch. The adsorbents were added to 100 mL of 1 ppm of heavy metals solution in a 250 mL conical flask at room temperature. All the samples were placed on a rotary shaker at 150 rpm. The samples will be withdrawn and filtered after 60 minutes. The metals solution was analysed with FAAS to determine the concentration of the residual heavy metals solution.

3.3.2. Effect of initial concentration

Weight of adsorbent was fixed at 1.0 g the rotary speed was fixed at 150 rpm for 60 minutes. The initial concentration of the metals solutions were varied 10, 20, 30, 40, and 50 ppm with 100 mL volume each. A blank control was prepared for every concentration of metals solution. The samples were filtered after 60 minutes and the metals solution was analysed with Flame Atomic Absorption Spectroscopy (FAAS) to determine the concentration of residual heavy metals solution.

3.3.3. Effect of agitation time

The agitation time that was used was 20, 30, 40, 50, and 60 minutes while the quantity of the adsorbent that will be used is 1.0 g with 150 rpm agitation speed, and initial ion concentration of 10 ppm with 100 mL volume. A blank control was prepared for every batch. The samples were filtered after agitated and the solution was analysed with FAAS to determine the concentration of residual heavy metals solution.

3.4. Removal efficiency

The removal efficiency was calculated by using Equation 3.1 below:

$$\text{Removal (\%)} = [(C_i - C_f)/ C_i] \times 100\% \qquad (3.1)$$

where,

C_i : the initial metal solution concentration (ppm)

C_f : the final metal solution concentration (ppm)

4. Results and Discussion

4.1. Adsorption study

4.1.1. Effect of adsorbent dosage

The adsorbent dosage is one of the crucial parameters to regulate both the availability and the accessibility of adsorption sites (Li, Du, Chen, & Zhang, 2007). The relationship between adsorbent dosage with the percentage of removal efficiency of the AC produced from pepper waste and CAC is shown in Figure 4.1, Figure 4.2 and Figure 4.3. The adsorbent dosage was varied from 0.2 g to 1.0 g under a constant condition (150 rpm agitation speed, 60 minutes at room temperature). Figure 4.1, Figure 4.2 and Figure 4.3 show that as the adsorbent dosage increased from 0.2 g to 1.0 g, the percentage removal also increased. Higher adsorbent dosage will increase the removal efficiency since more surfaces and functional groups available on adsorbent for metals to interact with (Wahi, Ngaini, & Jok, 2009). These chemical groups were crucial in the Van der Waals bonding formation since the functional groups was the main factor in binding metals to the adsorbent during adsorption (Malik, Ramteke, & Wate, 2007). There was a higher chance for the metals adsorption because there was less competition between metals for the binding sites as the adsorbent dosage increased. The removal efficiency of heavy metals by CAC showed higher percentage compared to pepper waste based AC as CAC can adsorb up to 92.71% of Fe, 22.30% of Zn and 99.70% of Pb whilst AC produced from pepper waste can only adsorb up to 33.96% of Fe, 16.88% of Zn and 78.37% of Pb. In this experiment, it can be shown that both types of ACs have different abilities in adsorbing different heavy metals as both ACs showed the same adsorption trends for the metals which is Pb>Fe>Zn. The same trends for metals adsorption was obtained by Bernard, Jimoh, and

Odigure (2013) in their study of heavy metals removal from industrial wastewater using coconut shell based AC on the same parameters which is adsorption of Pb was the highest and followed by Fe and then Zn.

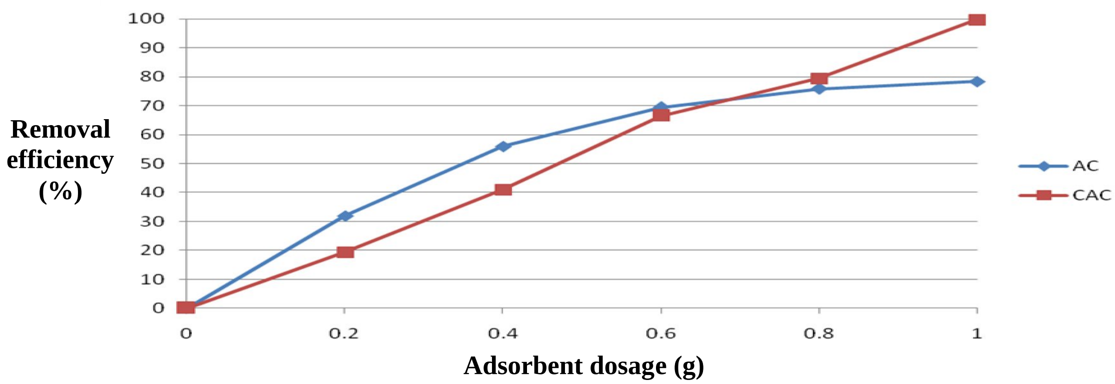

Figure 4.1. Effect of adsorbent dosage on Pb adsorption.

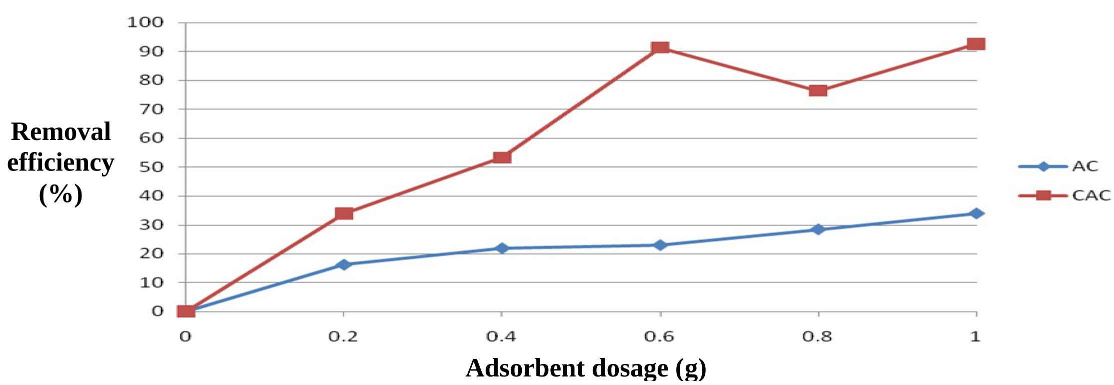

Figure 4.2. Effect of adsorbent dosage on Fe adsorption.

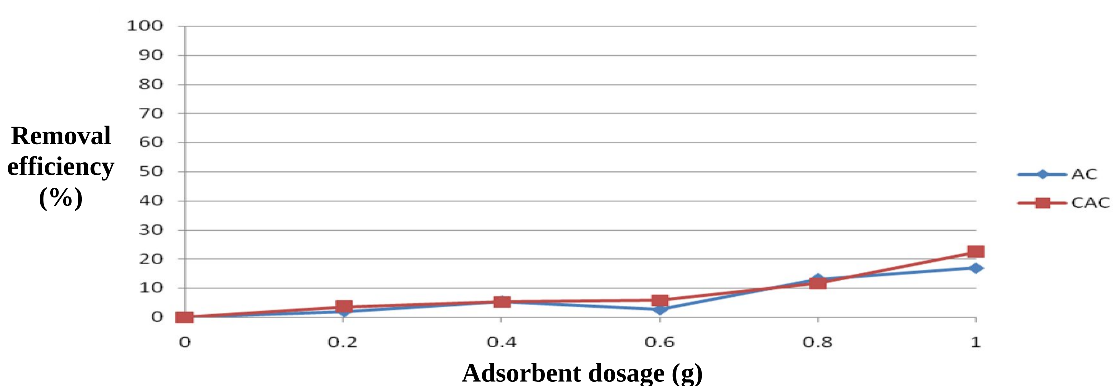

Figure 4.3. Effect of adsorbent dosage on Zn adsorption.

4.1.2. Effect of initial concentration

Figure 4.4, Figure 4.5 and Figure 4.6 shows the relationship between initial concentration of Pb, Fe and Zn with percentage of removal efficiency, respectively. The initial concentration of heavy metals solution was varied from 10 ppm to 50 ppm with constant adsorbent dosage of 1.0 g and 60 minutes contact time at 150 rpm agitation speed. It can be seen that the percentage of removal efficiency decreased as the initial concentrations of Pb, Fe and Zn increased (Figure 4.4, Figure 4.5 and Figure 4.6). This can be cause by the higher availability of adsorption sites on the adsorbent at low concentration of metals solution (Qian, Machida, & Tatsumoto, 2008). The decreased in percentage of removal efficiency of metals can be due to the fact that the sites for adsorption at the adsorbent surface becomes more saturated as the initial metals concentration was increased but the amount of adsorbent was maintained (Bhatti, Mumtaz, Hanif, & Nadeem 2007). AC produced from pepper waste showed higher ability in adsorbing Pb and Zn compared to CAC. Contrary results was showed for Fe where the CAC adsorb higher than pepper waste based AC. Both pepper waste based AC and CAC showed the highest percentage of adsorbing Pb metals which is 98.93% and 94.20%, respectively at the concentration of 10 ppm. These percentage of removal efficiency displayed minimal difference compared to palm oil empty fruit bunch based AC where the removal of Pb metals has reached 100% in between 5 to 20 ppm of metal solutions initial concentration (Wahi, Ngaini, & Jok, 2009). From Figure 4.4 and Figure 4.5, it can be seen that further increase in the initial concentrations did not bring any further improvement for the metals adsorption but it resulted in desorption of the metals from the adsorbent surface.