

THE REMOVAL OF PLUMBUM AND ZINC USING ACTIVATED CARBON

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DECLARATION

I declare that this thesis entitle “The removal of plumbum and zinc using activated carbon” is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :.....

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Date : May 14th, 2008

DEDICATION

*Special dedication to my family members,
My friends, my fellow colleague
and all faculty members
For all your Care and Support*

*Sincerely,
Asong Angkat*

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In preparing this thesis, I was contact with many people, academicians, researchers and practitioners. They have contributed toward my understanding and thoughts during this project development. In particular, I wish to express my sincere appreciation to my thesis supervisor, Mr Mior Ahmad Khushairi bin Mohd Zahari for his encouragement, guidance, critics and advise. Without his continued support and interest, this thesis would not be a success.

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ABSTRAK

Eksperimen penyingkiran logam berat menggunakan karbon teraktif ini dijalankan bertujuan mengkaji kesan kepekatan karbon teraktif dan pH ke atas penyingkiran logam berat. Dalam kajian ini, perbezaan antara saiz karbon teraktif turut dikaji. Eksperimen ini dilakukan dengan menggunakan sukatan karbon teraktif yang berbeza untuk mengkaji kesan perbezaan kepekatan. Ia dilakukan dengan mencampurkan sukatan karbon teraktif yang berbeza ke atas larutan logam. Di samping itu, kajian juga dilakukan pada nilai pH yang berbeza dengan menambah larutan logam berat yang mempunyai pH berbeza ke atas sukatan karbon teraktif bagi mendapatkan penyingkiran logam berat yang maksimum. Karbon teraktif berbentuk serbuk dan granular digunakan untuk melihat kesan perbezaan saiz. Campuran tersebut digoncang dengan menggunakan "kelalang goncang" selama sejam pada suhu 30°C. Sampel diambil setelah campuran digoncang dan dianalisis dengan menggunakan alat "Spectrofotometer Serapan Atom (AAS)". Daripada keputusan yang diperolehi, didapati bahawa peratus penyingkiran kedua-dua ion logam adalah paling tinggi pada pH 10 iaitu, 92.8% bagi ion zink dan 93.36% bagi ion plumbum. Kuantiti dan saiz karbon teraktif yang digunakan juga turut memberi pengaruh yang besar dalam kadar penyingkiran ion logam berat. Daripada keputusan yang diperolehi, didapati bahawa penyingkiran ion logam adalah paling tinggi apabila sebanyak 5g karbon teraktif dimasukkan ke dalam larutan air sisa bagi kedua-dua ion logam. Peratus penyingkiran ion logam yang diperolehi ialah sebanyak 47.2% bagi ion zink dan 62.68% bagi ion plumbum. Peratus penyingkiran ion plumbum ialah sebanyak 95.2% dengan menggunakan karbon teraktif dalam bentuk serbuk manakala 53.74% bagi karbon teraktif dalam bentuk granular. Kesimpulannya, kajian ini menunjukkan bahawa kuantiti karbon teraktif, saiz karbon teraktif dan pH logam berat amat memberi kesan kepada penyingkiran ion logam berat.

ABSTRACT

This project was about removing heavy metal, Pb and Zn using activated carbon and to investigate the effect of activated carbon dosage and pH of heavy metal solution during the experiment. In the experiment, the effect of different particle size of activated carbon also being studied. The experiment was using different doses of activated carbon to investigate the removal of heavy metal. It was carried by adding different doses of activated carbon to stock solution. Besides, it was also tested at different value of pH, which was done by adding an amount of activated carbon that gives the best performance into different pH of stock solution to get the maximum heavy metal removal. The powdered and granular activated carbon (GAC) used to see the different effect. To get the maximum effect, the solution was well mixed using the orbital shaker for 1 hour at 30°C. Samples were taken after it mixed and analyzed using Atomic Absorption Spectrophotometer (AAS). From the result, pH 10 gives the highest removal percentage of the heavy metal which is 92.8% for zinc ion and 93.36% for plumbum ion. From the result also, the removal percentage of heavy metal ion was highest when 5g of activated carbon was added to both simulated waste water. The removal percentage was 47.2% for zinc ion and 62.68% for plumbum ion. The result also shows that the powdered activated carbon (PAC) can increase the percentage of heavy metal ion removal compare to GAC. The removal percentage was 95.2% when the PAC was used while 53.74% for GAC. As conclusion, we know that activated carbon dosage and pH totally influence the removal of heavy metal ion.

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LIST OF ABBREVIATION

| SYMBOL | DIMENSION |
|---------------|---------------------|
| °C | degree Celsius |
| °F | degree Fahrenheit |
| g | gram |
| cm | centimeter |
| cal | calory |
| s | second |
| K | kelvin |
| μ | viscosity |
| Ω | ohm |
| % | percent |
| pH | potential hydrogen |
| cc | Cubic capacity |
| rpm | Rotation per minute |
| ml | mililiter |
| ppm | Part per million |

CHAPTER 1

INTRODUCTION

1.1 Background of study

The removal of heavy metal contaminants from aqueous solutions is one of the most important environmental concerns because metals are biorefractory, and are toxic to many life forms. Various sources of lead and zinc in water are battery manufacturing, basic steel, paper and pulp, metal plating, leather tanning, agrochemicals, petrochemicals and fertilizer industries. Higher concentrations of heavy metals in water and soils may increase the uptake of these metals by crops and potentially affect human health via food chains. Lead is ubiquitous in the environment and is hazardous at high levels. It is a general metabolic poison and enzyme inhibitor (Li, 2002), causes toxicity to the neuronal system, and affects the function of brain cells (Ho, 2001). General symptoms of zinc toxicity are retardation of growth in plants (Vishwanadhan, 2000) and zinc fever and edema of lungs in human beings (Gupta and Sharma, 2003). Because of the above factors, there is a necessity to remove these metals from wastewater in order to prevent contamination of natural water bodies by effluents containing toxic metals. The common methods for removing metal ions from water and wastewater include chemical precipitation, oxidation, reduction, reverse osmosis, membrane filtration and adsorption. Among these, adsorption is effective and economical.

Activated carbon is one of the most popular adsorbents for the removal of metal ions from aqueous solutions (Kikuchi, 2006, Krishnan and Anirudhan, 2003, Machida, 2005, Malik, 2002 and Sekar, 2004). The high cost of commercially available activated carbon limits its use as an adsorbent in developing countries. Hence, there is a growing

need to prepare activated carbon from cheaper and locally available waste materials. Several researchers have used different activated carbons prepared from agriculture wastes such as oil palm shells (Kassim, 2004), coir pith carbon (Kadirvelu and Namasivayam, 2000), peanut husks (Ricordel, 2001), olive pulp (Galiatsatou, 2002) and charcoal (Mishra and Chaudhary, 1994) for the removal of lead and zinc from water and wastewater. There is still a need to develop activated carbon from cheaper and readily available materials, which can be effective and economical for the removal of lead and zinc from aqueous solutions.

In the present investigation, the potential of activated carbon for the removal of lead and zinc from aqueous solutions has been studied.

The studies relating to the removal of Pb and Zn from aqueous solutions using activated carbon have been carried out by batch method. Pb and Zn solutions in reagent bottles with activated carbon were shaken at different pH conditions for different time intervals. The remaining metal concentrations in aqueous solutions were measured by AAS method. The concentrations of metals and doses of activated carbon were varied. The particle size of activated carbon also was varied.

1.2 Objective of research

The objective of the experiment is to investigate the performance of activated carbon in the removal of plumbum and zinc by studying the activated carbon dosage ,pH of heavy metal and particle size of activated carbon.

1.3 Scope of studies

The scopes of this experiment are;

- (a) To study the effect of activated carbon dosage in the removal of ion zinc and plumbum.
- (b) To study the effect of pH on removal of zinc and plumbum.
- (c) To study the different partical size of activated carbon on zinc and plumbum.

1.4 Problem statement

Last few decades, heavy metal discharge into aquatic ecosystems has becoming a matter of concern. Heavy metals can enter a water supply by industrial and consumer waste, or even from acidic rain breaking down soils and releasing heavy metals into streams, lakes, rivers, and groundwater (<http://www.lenntech.com/periodic-chart-element.htm>). It is consist of mercury (Hg), cadmium (Cd), arsenic (As), chromium (Cr), thallium (Tl), and lead (Pb).The heavy metal especially zinc and plumbum that have been used in varies field in industry is chosen in this research. Plumbum is a highly toxic substance, exposure to which can produce a wide range of adverse health effects. Both adults and children can suffer from the effects of plumbum poisoning, but childhood plumbum poisoning is much more frequent. Over the many years since we have known about the hazards of lead, tens of millions of children have suffered its health effects. Even in 2004, there are still at minimum more than four hundred thousand children under the age of six who have too much lead in their blood (<http://www.nsc.org/library/fact/lead.htm>). While Zinc toxicity has been seen in both acute and chronic forms. Intakes of 150 to 450 mg of zinc per day have been associated with low copper status, altered iron function, reduced immune function, and reduced levels of high-density lipoproteins (the good cholesterol). One case report cited severe nausea and vomiting within 30 minutes after the person ingested four grams of zinc gluconate (570 mg elemental zinc). It is harmful if it is taken above suitable dose (<http://ods.od.nih.gov/factsheets/cc/zinc.html#issues>).

So, many research has been carried out in the area of activated carbon adsorption during the past four or five decades, and research data are scattered in different journals. Activated carbon is used in order to purify, decolorize, deodorize, dechlorinate, separate and concentrate to permit recovery and to filter, remove, or modify the harmful constituents from gases and liquid solutions. Consequently, activated carbon adsorption is of interest to many economic sectors and concern areas as diverse as food, pharmaceutical, chemical, petroleum, nuclear, automobile, and vacuum industries as well as for the treatment of drinking water, industrial and urban waste water, and industrial flue gases.

Activated carbon is chosen to be a method to remove zinc and lead because it is highly used in the industry. Activated carbon also has many advantages due to its usage. Lead and zinc are chosen to be the simulated removed heavy metal because of their harm and toxicity to the environment and human being. Lead and zinc also highly release to the environment. So, to prevent the environment from becoming more polluted by lead and zinc, it would be better to remove them using activated carbon.

CHAPTER 2

LITERATURE REVIEW

2.1 Absorption

Absorption, in chemical is a physical or chemical phenomenon or a process in which atom, molecules, or ions enter some bulk phase -gas, liquid or solid material. This is a different process from adsorption, since the molecules are taken up by the volume, not by surface. A more general term is sorption which covers adsorption, absorption, and ions exchange (Weber and Walter J, 1972). If absorption is a physical process not accompanied by any other physical or chemical process, it usually follows the Nernst partition law:

The value of constant K_N depends on temperature and is called partition coefficient. This equation is valid if concentrations are not too large and if the species "x" does not change its form in any of the two phases "1" or "2". If such molecule undergoes association then this equation still describes the equilibrium between "x" in both phases, but only for the same form - concentrations of all remaining forms must be calculated by taking into account all the other equilibria (Weber and Walter J, 1972).

In the case of gas absorption, one may calculate its concentration by using the Ideal gas law, $c = p/RT$. Alternatively, one may use partial pressure instead of concentrations.

In many technologically important processes, the chemical absorption is used in place of the physical process, e.g. absorption of carbon dioxide by sodium hydroxide - such processes do not follow the Nernst partition law (Weber and Walter J, 1972).

2.2 Adsorption

2.2.1 Adsorption Processes

Adsorption involves the interphase accumulation of concentration of substances at a surface or interface. The processes can occur at an interface between any two phases, such as, liquid-liquid, gas-liquid, or liquid-solid interfaces. Adsorbate is the material being concentrated or adsorbed while adsorbent are the adsorbing phase. Sorption, which includes both adsorption and absorption, is a general expression for a process in which a components move from one phase to be accumulated in another, particularly for cases in which the second phase is solid. There are relationship between surface tension and adsorption. Adsorption is a process in which matter is extracted from one phase and concentrated at the surface of a second - a surface phenomenon. Surface reactions of this type must occur at least partly as a result of the forces within the phase boundaries or surface boundaries.

Adsorption involves 3 types, which are exchange adsorption, physical adsorption and chemical adsorption. Exchange adsorption is when Ions of one substance concentrate a surface as a result of electrostatic attraction. The charge on the ion is the determining factor (trivalent ion is attracted much more strongly toward a site of opposite charge). Physical adsorption is a result of van der Waals force. The adsorbed molecule is not affixed to a specific site at the surface but is free the undergo translational movement within the interface. It is predominant at low temperature, and is characterized by a relatively low energy of adsorption. Chemical adsorption is the adsorbate undergoes chemical interaction with the adsorbent. (activated adsorption or chemisorption). It is favored by higher temperature, because chemical reactions proceed more rapidly at elevated temperatures. It exhibits high energy of adsorption. Most adsorption phenomenon is combinations of the three forms adsorption (Weber and Walter J, 1972).

2.2.2. Rates of Adsorption

There are three consecutive steps in the adsorption of materials from solution by porous adsorbents such as granular activated carbon.

- a. The transport of the adsorbate through a surface film to the exterior of the adsorbent.
- b. Sorption by porous adsorbents.
- c. Adsorption of the solute on the interior surfaces bounding the pore and capillary spaces of the adsorbent.

Under certain operating conditions, the transport of the adsorbate through the "surface film" or boundary layer to the adsorbent is the rate-limiting step (continuous flow systems through carbon beds). If sufficient turbulence is provided, transport of the adsorbate within the porous carbon controls the rate of uptake (batch reactor with a high degree of agitation) (Weber and Walter J, 1972).

2.2.3 Factors Influencing Adsorption

2.2.3.1 Surface Area

The extent of adsorption is proportional to specific area. Specific area can be defined as that portion of the total area that is available for adsorption (Weber and Walter J, 1972).

2.2.3.2 Nature of the Adsorbate

a. Solubility

Adsorption of a solute is inversely proportional to its solubility in the solvent. The greater the solubility, the stronger the solute-solvent bond and the smaller the extent

of adsorption (Lundelius's rule). In general, the solubility of any organic compound in water decreases with increasing chain length, because the compound becomes more hydrocarbon-like as the number of carbon atoms becomes greater (Traube's rule). Adsorption of aliphatic series of organic acids increases in the order formic-acetic-propionic-butyric, whereas the order is reversed for adsorption from toluene (Weber and Walter J, 1972).

b. Molecular sizes.

If the rate is controlled by intraparticle transport, the reaction generally will proceed more rapidly the smaller the adsorbate molecule, within a given class of compound (Weber and Walter J, 1972).

c. Charge (Ionic species):

As long as the compounds are structurally simple adsorption is at minimum for the charged species and at a maximum for the neutral species. As compounds become more complex, the effect of ionization becomes of decreasing importance. Decreasing adsorption with increasing ionization has been observed for many organic acids. Adsorption of propionic acid on carbon decreases markedly with increasing pH to a minimum in the range pH 3.5 to 5.5 (succinic acid to a min. in the range pH 4.0 and 7.0, caproic acid between pH 4.8 and 7.5). Studies on amphoteric compound indicate an adsorption maximum at the isoelectric point, or that pH at which both the acidic end and the basic end of the compound are ionized and the compound bears a net charge of zero (Weber and Walter J, 1972).

d. pH

Because hydrogen and hydroxide ions are adsorbed quite strongly, the adsorption of other ions is influenced by the pH of the solution. In general, adsorption of typical organic pollutant from water is increased with decreasing pH (Weber and Walter J, 1972).

e. Temperature

Adsorption reactions are normally exothermic, thus the extent of adsorption generally increases with decreasing temperature (Weber and Walter J, 1972).

f. Adsorption of Mixed Solutes

The degree of mutual inhibition of competing adsorbates should be related to the relative sizes of the molecules being adsorbed, to the relative adsorptive affinities, and to the relative concentrations of solutes (Weber and Walter J, 1972).

g. Nature of the Adsorbent

The physicochemical nature of the adsorbent can have profound effects on both rate and capacity for adsorption. Commercial carbons can be prepared from a variety of raw materials, including wood, lignite, coal, petroleum residues, and nut shells. The raw material is generally activated in an atmosphere of CO₂, CO, O₂, H₂O, vapor, air, or other selected gases, at a temperature between 300 and 1000°C often followed by quenching in air or water. The most characteristic physical property of activated carbon is its extremely large surface area, which is composed mainly of surfaces bordering inner pore spaces (Weber and Walter J, 1972).

2.3 Activated carbon

Activated carbons are versatile adsorbents. Their adsorptive properties are due to their high surface area, a microporous structure, and high degree of surface reactivity. They are, used, therefore, to purify, decolorize, deodorize, dechlorinate, separate, and concentrate in order to permit recovery and to filter, remove, or modify the harmful constituents from gases and liquid solutions. Activated carbons have a highly developed porosity and an extended interparticulate surface area. Their preparation involves two main steps: the carbonization of the carbonaceous raw material at temperature below 800 °C in an inert atmosphere and the activation of the carbonized product. Thus, all

carbonaceous materials can be converted into activated carbon, although the properties of the final product will be different, depending on the nature of the raw material used, the nature of the activating agent, and the conditions of the carbonization and activation processes (Roop C.B and Meenakshi G, 2002).

During carbonization process, most of the noncarbon element such as oxygen, hydrogen, and nitrogen are eliminated as a volatile gaseous species by the pyrolytic decomposition of the starting material. The residual elementary carbon atoms group themselves into stacks of flat, aromatic sheets cross-linked in a random manner. These aromatic sheets are irregularly arranged, which leaves free interstices. These interstices give rise to pores, which make activated carbons excellent adsorbent. During carbonization these pores are filled with the tarry matter or the product of decomposition or at least blocked partially by disorganized carbon. This pore structure in carbonized char is further developed and enhanced during the activation process, which converts the carbonized raw material into a form that contains the greatest possible number of randomly distributed pores of various sizes and shapes, giving rise to an extended and extremely high surface area of the product (Roop C.B and Meenakshi G, 2002).

Activated carbon has a microcrystalline structure. But this microcrystalline structure has interlayer spacing with range between 0.34 and 0.35nm in activated carbon. The orientation of the stacks of aromatic sheet is also less ordered in activated carbons. The aromatic sheet contains free radical structure or structure with unpaired electrons (Roop C.B and Meenakshi G, 2002).

The activated carbon in general have strongly developed internal surface and are usually characterized by a polydisperse porous structure consisting of pores different sizes and shapes. Because the pores have walls, they will comprise two types of surface: the internal or microporous surface and the external surface. The former represents the wall of the pores and has high surface area that may be several thousand in many activated carbons. Besides the crystalline and porous structure, an activated carbon surface has a chemical structure. The adsorption capacity of an activated carbon is

determined by the physical or porous structure but strongly influence by chemical structure of the carbon surface (Roop C.B and Meenakshi G, 2002).

Carbon-oxygen surface groups are by far the most important surface group that influences the surface characteristic such as the wettability, polarity, and acidity, and the physico-chemical properties such as catalytic, electric, and chemical reactivity of these materials. In fact, the combined oxygen has often found to be the source of the property by which a carbon becomes useful and effective in certain respects. The surface groups can not be treated as ordinary organic compounds because they interact different environments (Roop C.B and Meenakshi G, 2002).

2.4 Heavy metal

2.4.1 Introduction

The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations. Examples of heavy metals include mercury (Hg), cadmium (Cd), arsenic (As), chromium (Cr), thallium (Tl), and lead (Pb).

Heavy metals are natural components of the Earth's crust. They cannot be degraded or destroyed. To a small extent they enter our bodies by food, drinking water and air. As trace elements, some heavy metals like copper, selenium and zinc are essential to maintain the metabolism of the human body. However, at higher concentrations heavy metal can lead to poisoning. Heavy metal poisoning could result, for instance, from drinking-water contamination, high ambient air concentrations near emission sources, or intake via the food chain.

Heavy metals are dangerous because they tend to bioaccumulate. Bioaccumulation means an increase in the concentration of a chemical in a biological organism over time, compared to the chemical's concentration in the environment.

Compounds accumulate in living things any time they are taken up and stored faster than they are broken down (metabolized) or excreted.

Heavy metals can enter a water supply by industrial and consumer waste, or even from acidic rain breaking down soils and releasing heavy metals into streams, lakes, rivers, and groundwater.

2.4.2 Plumbum

The symbol in the title is the alchemical symbol for plumbum (lead), the sign of Saturn. Lead is a shiny, blue-white soft metal when its surface is fresh. On exposure to the air, it becomes covered by a dull, gray layer of basic carbonate that adheres closely and protects it from further alteration. It resembles aluminium in this respect, which is protected by a dull, gray layer of oxide. Otherwise, lead would react rapidly with the oxygen and carbon dioxide in the air. When placed in sulphuric acid, lead is protected by a similar layer of PbSO_4 that adheres strongly. For these reasons, lead is often used to sheathe cables for burial, to protect roofs from the atmosphere, and as tanks and pipes for sulphuric acid. Lead has many and varied uses in technology. This article mentions a very large number of uses of lead and its compounds, from cathedrals to crystal sets, from batteries to sailing ships (<http://www.du.edu/~jcalvert/phys/lead.>).

Lead and its compounds were known and widely used in antiquity. Its metallurgy was well-developed even then. The uses of lead depended on its corrosion resistance, its softness and ease of working, and its low melting point. Buildings were roofed with sheets of lead, and glazed with lead mullions. Molten lead was poured into holes in stone to hold fasteners. Boxes of lead were used as protective containers, and as coffins. Lead was used in the metallurgy of other metals, as we shall see in some detail. Lead was, indeed, used in machines of all kinds, not as a structural material, but where its fusibility and workability were an advantage, and for many small items of daily use (<http://www.du.edu/~jcalvert/phys/lead.>).

At the current time, the greatest quantity of lead is used in lead-acid storage batteries. Until recently, next in importance was the use in paint pigments. However, environmental considerations have largely removed lead from paints. Similarly, the once considerable usage of lead in tetraethyl lead antiknock compound has also disappeared for a similar reason. Lead is still used for sheathing cables, in bearing alloys, in artistic pigments and glazes, for decorative glass, in the chemical industry, and, of course, for bullets, which have always been in special demand in the United States (<http://www.du.edu/~jcalvert/phys/lead.>).

2.4.2.1 Properties of Plumbum

Table 2.1: Properties of plumbum

| Properties | Measurement |
|---------------------------------|--------------------------------|
| The atomic number | 82 |
| stable isotopes | 204, 206, 207 and 208 |
| average atomic weight | 207.22 g/mol |
| density | 11.3437 g/cm ³ |
| crystallizes | face-centered cubic structure |
| lattice constant | a = 0.494 nm |
| melting point | 327.35°C |
| boiling point | 1515°C |
| coefficient of linear expansion | 29.5 x 10 ⁻⁴ per °C |
| bulk modulus | 0.44 x 10 ⁶ Mbar |
| heat conductivity | 0.081 cal/cm-s-°C |
| specific heat | 0.03046 cal/g-K |
| heat of fusion | 6.26 cal/g |
| hardness | 1.5 on the Mohs scale |
| tensile strength | 2000 psi |
| Young's modulus | 2.56 x 10 ⁶ psi |