EFFECT OF pH ON THE ADSORPTION OF LEAD (II) ION BY DRIED CATTAIL LEAVES

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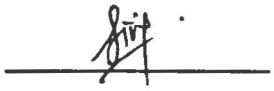

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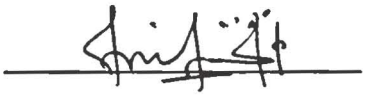

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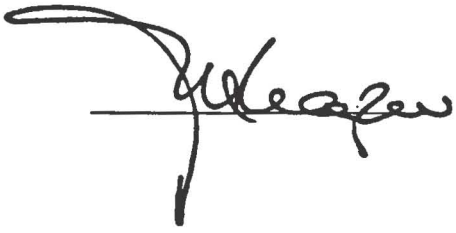

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ABSTRAK

Tujuan kajian ini adalah untuk mengkaji kesan pH terhadap jerapan, menentukan isoterma dan kinetik jerapan ion Pb (II) dengan menggunakan daun banat kering. Daun banat diambil dari tasik Kampung E, Universiti Malaysia Sabah. Daun Banat dibersihkan and dikeringkan. Experiment dijalankan dengan mengoncangkan daun banat kering dengan larutan ion Pb (II) pada pH yang berlainan. Sampel dituras dan dianalisis selepas mencapai masa sentuhan yang ditentukan. Jerapan ion Pb (II) bertambah apabila pH larutan bertambah. Kajian ini didapati mengikut isoterma Langmuir dengan kapasiti jerapan maksimum sebanyak 57.14 mg/g. Kinetik jerapan didapati mengikuti pseudo-kedua order kinetik. Ini menunjukkan jerapan kimia terlibat dalam jerapan ion Pb (II).

ABSTRACT

The objective of this study is to investigate the effect of pH on the adsorption, to determine isotherm and kinetic of adsorption of Pb (II) ions by dried cattail leaves. Cattail leaves was harvested from Kampung E Lake, Universiti Malaysia Sabah. Cattail leaves was clean and dried. Experiment was carried out by agitating dried cattail leaves with Pb (II) solution at different pH. Sample was filtered and analyzed after reach designated contact time. The adsorption of Pb (II) ions is increase as the pH is increase. This study followed Langmuir isotherm with maximum adsorption capacity 57.14 mg/g. The adsorption kinetic followed the pseudo-second order. This indicates that the chemisorption involved in adsorption of Pb (II) ions.

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C	Boundary layer thickness
C_e	Concentration of sorbate at equilibrium
C_o	Initial concentration of sorbate
g/mol	Gram per mol
h	Hour
HNO_3	Nitrate acid
k_1	Pseudo-first order kinetic model rate constant
k_2	Pseudo-second order kinetic model rate constant
K_{id}	Intraparticle diffusion rate constant
mg/g	Milligram per gram
min	Minutes
mL	Millilitre
NaOH	Sodium hydroxide
$Pb(NO_3)_2$	Lead nitrate
ppb	Part per billion
Q_e	Amount of metal ions adsorbed at equilibrium
Q_m	Maximum adsorption capacity
Q_t	Amount of metal ions adsorbed at time
R^2	Determination coefficient
R_L	Separation factor
rpm	Revolution per minutes
t	Time
w	Weight

CHAPTER 1

INTRODUCTION

Water is an essential component for living organisms on the Earth. Living organisms cannot sustain their live without water. The cell of living organisms is made up of at least 60% of water (Enger and Smith, 2004). In living organisms, water acts a medium for metabolic activity. It helps dissolve and transport nutrients, regulates body temperature, support structures and remove waste products. Beside that, water also required in human activities such as agriculture, industry, transportation, power generation and others.

Water use can divide mainly into domestic use, agricultural use, industrial use and in-stream use. According to Botkin and Keller (2005), the domestic use account for 10% of total national water withdrawals. Drinking, cooking, bathing, lawns, toilet flushing laundry and dishes are typical domestic water use. Agriculture is the largest user of water which accounts around two-third of human water use (Kaufmann and Cleveland, 2008). Usage of water in agriculture include in irrigation and livestock. Industrial is another user that uses water extensively. In industry, water use as raw material, coolant, solvent, transport agent, energy source etc. On the other hand, in-stream uses of water are for recreation, navigation and hydroelectric power.

Abundance of water can be found on the Earth as oceans, lakes, rivers, glaciers and other water body cover two-third of the Earth surface. Although the Earth surface is cover mainly by water yet the usable water is limited. This is because over 97% of the water is saltwater which is unsuitable for human use

especially for drinking purpose; 2% of water is locked up as glacier and the remaining 1% of water most of it is in inaccessible aquifers (Mackenzie, 2003). As a consequent, only tiny portion of water is available for human use.

Hydrological cycle is a biogeochemical cycle that responsible constantly in redistributes water in the environment. This cycle involve the process of transfer of water from oceans to the atmosphere to the land and back to the ocean. As water receives heat, it will evaporate from land, water, organisms or runoff and enter into the atmosphere. Water in atmosphere later condenses and precipitates to the earth surface. Precipitation water may infiltrate into soil or overland by runoff into rivers, lakes and seas. However, human activities had cause disturbance on this cycle and degrade the quality of water.

Water pollution can be defined as any physical, biological, or chemical change in water quality that adversely affects living organisms or makes water unsuitable for desired uses (Cunnigham and Saigo, 1999). Rapid economic development and increasing human population has deteriorated the water pollution problem. Increase in industry activities in order to supply human demand had introduces more pollutants into the water body and this turn further degrades the water quality. In addition, high water demand cause fast water withdrawal and groundwater become more easily depleted. As a result, lack of safe and potable water for human.

Water pollution has seriously affects the water supplies throughout the world (Enger and Smith, 2004). This problem had become major issue in both developed country and developing country. People unable to access safe water drinking and disease related to waterborne had been induced. Death also has induced in people who seriously suffer water-related disease.

Pollutants that had been induced by human activities include pathogens, heavy metals, sediments, heat, nutrient, radioactive isotope and others. Water

pollution by heavy metals is one of the pollution had been receiving great concern (Jaramillo *et al.*, 2009). According to Yu (2005), heavy metal is element with density greater than 5 g/cm^3 and atomic mass more than calcium. Heavy metals such as mercury, lead, cadmium, nickel, gold, platinum, silver, bismuth, arsenic, selenium, vanadium, chromium, and thallium are categorised as major heavy metal which can cause health hazard to human and ecosystems (Botkin and Keller, 2005).

The industries that had contributed heavy metal to the wastewater include metal finishing, electroplating, plastics, pigments, mining and others (Dang *et al.*, 2009). Most of the heavy metals are toxic, non-biodegradable and tend to accumulate in living organisms (Shek *et al.*, 2009). The bioaccumulation of heavy metal in human body may cause the damage of mental and central nervous functions, and harm the important organ such as lung, kidneys, livers etcetera.

Heavy metal from both natural and anthropogenic sources can become concentrated in living organisms through toxic pathway. This toxic pathway includes transfer of toxic in physical system such as atmosphere, land, water, and sediment. From physical system, the toxin concentration is increasing when it is transfer towards biological system such as plants, herbivores, carnivores and humans through biomagnifications.

There are increases in awareness of water quality. Rules and regulation has been implemented to reduce water pollution problem. Wastewater from industries has to undergo treatment process before discharge to the river or sea. By obeying certain law or enactment, only permissible level of pollutants is allowed to be discharge into sea or river. According to Clean Water Act of 1977 of United States, it is illegal to discharge pollution from a point source without a permit. It set standards for industrial wastewaters and funded construction of sewage treatment plants (Withgott and Brennan, 2007).

Water pollution by heavy metal had cause economy burden to a country. Huge amount of money is spent in invention of technology and treatment processes. Increase of water pollution problem will the increase the total amount of money spent in it. Beside money, time and effort also required in order to supply clean and potable water.

Heavy metal had caused threat on living organisms on the Earth. Yet, due to its technological importance, heavy metal still being used in industries activities (Febrianto *et al.*, 2008). As a result, the cost effective methods for the removal of heavy metals from aqueous solution become important issues of the world (Iftikhar *et al.*, 2009).

1.1 Objectives

In this work, dried cattail leaves were used as sorbent to adsorb Pb (II) ions from aqueous solution. The objectives of this research outline as follows:

(i) to investigate the effect of pH on the adsorption of Pb (II) ions by dried cattail leaves

(ii) to determine the adsorption isotherm for adsorption of Pb (II) ions by dried cattail leaves

(iii) to determine the kinetic study of the adsorption of Pb (II) ions by dried cattail leaves

CHAPTER 2

LITERATURE REVIEW

2.1 Removal of heavy metal

Heavy metal in the water and wastewater can be removed by various methods. These methods include chemical precipitation, ion exchange, reverse osmosis, adsorption and others. Table 2.1 showed the advantages and disadvantages of different heavy metal removal methods.

Table 2.1 Advantages and disadvantages of different heavy metal removal method.

Method	Advantages	Disadvantages	Reference
Chemical precipitation	Simple, inexpensive	Produces sludge, require large amount of chemical	Shukla *et al.*, 2005
Ion exchange	Suitable for low concentration	Expensive, non selective, sensitive to pH, unsuitable for highly concentrated solution	Ahluwalia and Goyal, 2006
Reverse osmosis	No addition of chemical, require low energy	High operational cost	Wang *et al.*, 2009
Adsorption	Simple, feasible, ease of operation	High cost of activated carbon	Ozcan *et al.*, 2009

Adsorption is highly effective, cheap and easy to adapt method among others method in removing of heavy metal (Eren *et al.*, 2009). Yet, due to high cost of activated carbon has limited its usage. Nevertheless, various agriculture wastes had been use as adsorbent in heavy metal removal (Malik *et al.*, 2004). This is because most of the agriculture waste is cheap and abundant in nature. The agricultural

wastes that had been employed include sawdust, rice hulls, coconut husk, pine bark, sugarcane bagasse and so on.

2.2 Lead

In the periodic table of elements, lead is in Group IVa. It has atomic weight 207.19 and atomic number 82. Its density is 11.34g mL^{-1}. The melting point for lead is 327.5 °C whereas boiling point is as high as 1749 °C. Lead is a soft metal with bluish white in colour and has three oxidation states: 0, +2, and +4. The dominant oxidation state of lead in most inorganic compounds is +2 (Merian *et al.*, 2004). Lead poses four natural Pb isotopes: ^{204}Pb, ^{205}Pb, ^{207}Pb and ^{208}Pb.

The chemical characteristics of lead include it oxidizes readily in the air under ordinary temperature. It is resistance to corrosion. Lead will combines will halogens, sulphur, and tellurium under heating condition. For the reaction with acids, lead does not react with dilute sulphuric acid, dilute hydrochloric acid, and hydrofluoric acid except dilute nitric acid (Filov *et al.*, 1993). This is due to the formation of protective layer on the surface of the sulphate, chloride and fluoride. There are varieties of metals forms alloy with lead.

Source of lead in the environment can be come from natural source and anthropogenic source. The natural source of lead is from the mineral galena (PbS) (Merian *et al.*, 2004). Other natural sources of lead include anglesite ($PbSO_4$) and cerrussite ($PbCO_3$) which is the natural weathering product of PbS. On the other hand, the anthropogenic sources are from the manufacturing of battery, automobile, ammunition, solders, caulking compounds, pigments, paints, herbicides, insecticides, radioactivity shields, plumbing fixtures etcetera.

Lead is widely used in industrial processes due to its ideal properties in industrial processes. For instance, lead is favour in industrial processes as it is easy to handle and shaped which is due to the softness and low melting point of lead. High density of lead makes it suitable as a shield against radiation and X-rays in nuclear industry and in hospitals, respectively. In addition, high density also makes lead effective in stopping sound waves. Thus, it is also apply in factories and ships to diminish noise from the machinery and engines. Characteristics of high resistance to corrosion make lead appropriate as material in weather proofing buildings and equipment of acids manufactured.

Lead can enter the environments through atmosphere, water body, soil, inhalation, skin contact and ingestion. However, the primary way of lead enter into human body is through inhalation and ingestion (Filov *et al.*, 1993; Cockerham and Shane, 1994; Hathaway and Protoc, 2004). Inhalation and ingestion of excess lead into the body will cause lead toxicity. Exposure of lead through inhalation include from workplace, cigarette smoking, indoor shooting, soldier moulding, exercise in lead-contaminated area and others. Due to their repeated hand-to-mouth activities, children are the groups of people who are more susceptible to exposure of lead through ingestion.

Lead toxicity had cause significant effect to plants, animals and humans. In plant, excess lead concentration will hinder the growth, metabolism and photosynthesis process. The effect of lead toxicity on animals and human body can range from gastrointestinal disturbances and anaemia, to more serious intoxification, neuromuscular dysfunction and to encephalopathy which is the most severe lead exposure.

Once a person is lead poisoning, he will experience these onset symptoms: weight loss, weakness, tiredness, insomnia and hypertension. In addition, there is also disturbance in his gastrointestinal tract. These disturbances in gastrointestinal tract include constipation, anorexia, abdominal discomfort and actual colic.

Hypochromic, normocytic type of anaemia is mostly happen in lead poisoning person where there is reduction in mean corpuscular haemoglobin as a result of stippling of erythrocytes and reticulocytosis (Hathaway and Protoc, 2004; Merian *et al.*, 2004).

However, the sign of neuromuscular dysfunctions such as motor weakness, paralysis of the extensor muscle of wrist and of fingers is more significant compared to illness symptoms mentioned above. Beside that, severe lead poisoning may lead to encephalopathy, a general term used to describe various illnesses which affect the brain function (Eren *et al.*, 2009). Encephalopathy is normally associated with ataxia, coma and convulsion.

Beside that, researches have showed that high occupational exposure lead level had cause health problems to the workers. There is mildly reduced in neuropsychological tests performance of lead workers. This is associated with non-specific subjective symptoms like anxiety, depressed mood, poor concentration, and forgetfulness in lead workers. On the other hand, there is increase in incidence of spontaneous abortions, miscarriages, and stillbirths for high occupational exposure lead levels in pregnant women (Hathaway and Protoc, 2004). Moreover, the kidney and reproductive system of both male and female is also being affected by lead poisoning.

Exposure to excess lead will cause damage in proximal tubular damage which consequently leads to the reversible Fanconi syndrome-like condition with aminoaciduria, glucosuria and hyperphosphaturia; and the leakage of the enzymes into the urine. The damage of kidney also can extend to irreversible nephropathy (Merian *et al.*, 2004). Toxicity of lead may damage the endocrine function of male where spermatogenesis and sperm motility is reduced, spermatozoa are affected, and testicular atrophy is induced. In female, lead poison will affect the hypothalamus- pituitary- ovarian- uterine function, change in menstrual cycle, implantation problems and so forth.

2.3 Adsorption

Adsorption is a surface process which can be defined as the accumulation of the molecules on the solid or liquid surface (Noll *et al.*, 1992). Sorbate is the substance that adsorb on the surface of solid or liquid and the place where the sorbate adsorb is known as adsorbent.

Adsorption process is significant to the environment as it can help to remove chemical substance and eliminate possible impact to living organisms. It is widely apply in both industrial and pollution control processes (Ucun *et al.*, 2008). The application of adsorption in pollution control processes include in control of organic compounds such as volatile organic compound and pesticide, and inorganic compound like heavy metal, chlorine, reduced sulphur gases etcetera.

Adsorption is divided into two types: multilayer adsorption and monolayer adsorption. Table 2.2 showed the comparison between multilayer adsorption and monolayer adsorption (Masel, 1996).

Table 2.2 Comparison between multilayer adsorption and monolayer adsorption.

Type of adsorption	Multilayer adsorption	Monolayer adsorption
Number of adsorbate layer found on adsorbent surface	Several layers	Single Layer
Interaction between adsorbate and adsorbent	Large	Small or direct
Temperature	Close to the boiling point of a adsorbate	Hundreds of degrees above the boiling point of adsorbate

In addition, adsorption can also be categorized into physical adsorption and chemisorption. According to Noll *et al.*, (1992), it is difficult to classify a system explicitly as most of adsorption processes are intermediate. Physical adsorption involves relatively weak intermolecular forces whereas strong chemical bonding is formed between sorbate and adsorbent surface in chemisorption. There is no

sharing or transfer of electron in physical adsorption and the interaction is reversible. This reversible interaction allow arise of desorption at the same temperature. Beside that, there is no site specific for adsorbed molecule where they are free to cover the entire surface. On the other hand, the interaction in chemisorption is irreversible and the chemisorbed molecules are fixed at specific sites.

2.3.1 Adsorption isotherm

Adsorption isotherm is used to describe the interaction of metal ions with sorbent (Sangi *et al.*, 2008). It explains the distribution of solute in the solid phase and liquid phase at equilibrium conditions (Hawari and Mulligan, 2006). The experimental results are substitute into equilibrium isotherm model equation in order to determine the most suitable model for the sorption system (Shek *et al.*, 2009).

There are various isotherm models available which include Langmuir model, Freundlich model, Temkin isotherm, Florry-Huggins isotherm, Halsey isotherm, Dubinin-Radshkevich equation, Brunaeur-Emmer-Teller (BET) model, Sips isotherm, Redlich-Paterson isotherm and Toth equation (Febrianto *et al.*, 2008). However, the most common used two isotherms are Freundlich and Langmuir model. Equation of both models is showed in Appendix A.

In 1906, Freundlich developed Freundlich isotherm, an empirical equation which assume multilayer adsorption and heterogeneous sorption surface (Anayurt *et al.*, 2009). It assumed that the adsorption energy of metal ions binding on a sorption surface of sorbent depend on whether or not the occupied adjacent sites (Sangi *et al.*, 2008).

Langmuir isotherm or also known as monolayer adsorption was developed by Langmuir in 1918 (Dang *et al.*, 2009). There are three assumptions that must be

followed in Langmuir isotherm. These assumptions include monolayer adsorption, the entire surface sites are similar and only occupy by one adsorbed atom, and the ability of a molecule to be taking up by a given sites is independent of its neighbouring sites occupancy (Febrianto *et al.*, 2008).

2.3.2 Adsorption kinetics

Adsorption kinetic depicts the solute uptake rate and control the residence time of solute uptake at the solid-solution interface (Noeline *et al.*, 2005). Kinetic models describe the process of removal of metal ions from aqueous solution and help determine the rate-controlling mechanism of adsorption process either involved chemical reaction, diffusion control or mass transfer which in turn provides useful information in designing the adsorption operation (Gupta and Babu, 2009). Various kinetic models have been applied to the experimental data in order to predict the adsorption kinetics. These kinetic models include intraparticle diffusion, pseudo-first order, pseudo-second order kinetic model and others. Equation of these three models is showed in Appendix B.

Processes that controlled the sorption rate include the diffusion of solute from the solution to the film surrounding the particle, diffusion from the film to the particle surface (external diffusion), diffusion from the surface to the internal site (surface or pore diffusion) and uptake that may involve several mechanism such as physio chemical sorption, chemical sorption precipitation and others (Igwe and Abia, 2007). A straight line from the plot of amount of metal ions adsorbed on sorbent (Q_t) against square root of time ($t^{1/2}$) will be obtained if the intraparticle diffusion is involved in adsorption process. The rate controlling step is the only intraparticle diffusion as the line pass through the origin (Hameed *et al*, 2008).

Pseudo first-order kinetic model assumes that the rate of occupation of adsorption site is proportional to the number of unoccupied sites (Iftikhar *et al.*,

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