EFFECT OF INITIAL CONCENTRATION ON THE ADSORPTION OF COPPER (II) AND LEAD (II) IONS BY DRIED WATER LILY LEAVES

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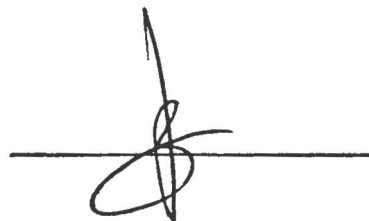
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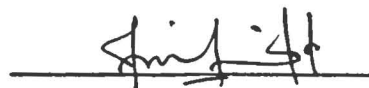# CERTIFICATION

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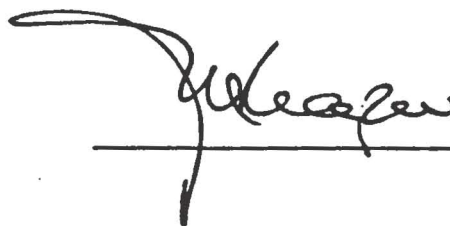2. **EXAMINER 1**

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ABSTRAK

Dalam kajian ini, kesan kepekatan awal dan masa sentuhan pada penjerapan ion Cu (II) dan Pb (II) dengan menggunakan daun teratai yang kering telah dikaji. Kajian ini dijalani dalam kaedah jerapan kelompok. Parameter-parameter seperti kepekatan awal dan masa sentuhan telah dianalisis dalam eskperimen ini. Keputusan kajian ini menunjukkan bahawa penjerapan Cu^{2+} dan Pb^{2+} adalah bergantung kepada kepekatan awal dan masa sentuhan. Kuantiti Cu^{2+} dan Pb^{2+} dijerap meningkat dengan peningkatan kepekatan awal dan masa sentuhan. Selain itu, penjerapan Cu^{2+} bersesuaian dengan isoterma Langmuir manakala penjerapan Pb^{2+} didapati bersesuaian dengan isotherma Freundlich. Kapasiti maksimum jerapan, Q_m, untuk Cu^{2+} dan Pb^{2+} adalah 17.85 dan 69.44 mg/g masing-masing. Tambahan pula, data eksperimen mengikuti model kinetik pseudo-kedua. Ini menunjukkan bahawa proses jerapan dikawal oleh jerapan kimia.

ABSTRACT

In this study, effect of initial concentration and contact time on the adsorption of Cu (II) and Pb (II) ions by dried water lily leaves was investigated. The research was carried out in batch adsorption experiment. Parameters such as initial concentration and contact time were studied in this experiment. The results of the study showed that the adsorption of Cu (II) and Pb (II) ions were dependent on initial concentration and contact time. The amount of Cu^{2+} and Pb^{2+} being adsorbed increases with increasing initial concentration and contact time. Next, the adsorption of of Cu^{2+} was found fitted with Langmuir isotherm whereas the adsorption of Pb^{2+} showed good agreement with Freundlich isotherm. The maximum adsorption capacity, Q_m, for Cu^{2+} and Pb^{2+} was 17.85 and 69.44 mg/g, respectively. Furthermore, experimental data was fitted into pseudo-second order kinetic model. This indicated that the adsorption process was controlled by chemical sorption.

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LIST OF SYMBOLS

Ø	Diameter
C_e	Concentration of sorbate at equilibrium
Q_e	Adsorption capacity at equilibrium
m	Mass
h	Initial adsorption rate
k_2	Pseudo-second order rate constant of adsorption
K_d	Intraparticle diffusion rate constant
K_F	Freundlich adsorption constant
K_L	Langmuir adsorption constant
n	Freundlich intensity constant
Q_e	Adsorption capacity at equilibrium
Q_m	Maxiumum adsorption capacity
Q_t	Amount of metal ion removed at particular time
R^2	Regression constant
R_L	Separation factor
S	Surface area for mass transfer
t	Time
β	Surface coverage constant
β_L	Liquid-solid mass transfer coefficient
α	Initial adsorption rate

LIST OF ABBREVIATIONS

FAAS   Flame atomic adsorption spectrometer

PP   Polypropylene

rpm   Revolution per minute

CHAPTER 1

INTRODUCTION

Most heavy metals are toxic even at relatively low concentrations, causing a variety of environmental problems (Botkin & Keller, 2005). Examples of heavy metal are lead (Pb), cadmium (Cd), nickel (Ni), copper (Cu), mercury (Hg), zinc (Zn) and arsenic (Ar). Heavy metal does not degrade into harmless end-product and tends to accumulate in living organisms (Imamoglu & Tekir, 2007). The heavy metal ions have become an eco-toxicological hazard of prime interest and increasing significance due to their accumulation nature in living organisms (Rao et al., 2005).

The presence of heavy metal in aquatic environment has become a great environmental concern to the world. Industries such as metal plating, mining, painting, car manufacturing, smelters and metal refineries are major sources of heavy metal pollution (Sangi et al., 2007). In Malaysia, release of industrial effluents containing toxic metals into the receiving water bodies is a major environmental concern and the discharges of wastewaters containing chemicals and metallic ions into nearby watercourses have been reported (Quek et al., 1998).

The removal of toxic metals from industries effluent so that their residual concentrations conform to the standards is a statutorily mandatory (Srivastava et al., 2007). The treatment for removal of metals from wastewaters can be done through precipitation, membrane filtration, ion exchange, adsorption and reverse osmosis (Kardirvelu et al., 2000).

As many conventional water treatment methods do not seem to be economically feasible due to their high operational costs, particularly during removal of metal ions at low concentrations (< 100 mg/L), adsorption has become one of the most popular methods and is currently considered as an effective, efficient and economic method for water purification (Sangi *et al.,* 2007). It is used as tertiary treatment process in wastewater treatment plants (Weng *et al.*, 2006). In adsorption, a substance is transferred from liquid phase to the surface of a solid and bounded by physical or chemical interactions (Kurniawan *et al.*, 2005). The ability of adsorption to remove toxic chemicals without disturbing the water quality has augmented its usage in comparison to electrochemical, biochemical or photochemical degradation processes (Mittal, 2005). Activated carbon is the most widely used adsorbent all over the world due to its high surface area, porous structure and special surface reactivity (Qu, 2008).

Aquatic macrophytes are aquatic photosynthetic plants which large enough to be seen with naked eyes. Generally, they can be classified into three categories which are submerged, floating or emerged plant species (Wang *et al.*, 1997). Aquatic macrophytes are beneficial to lake as they provide food and settler for fish and aquatic invertebrates. The vascular aquatic plants also act as biological filters of an aquatic environment (Vardanyan & Ingole, 2005). They help in removal of heavy metal by surface adsorption and incorporate the metal ions into their own system or store in a bound form (Rai *et al.*, 1995). The uptake of metal of aquatic plant depends on the type of metal and the species of plant (Espinoza-Quinones *et al.*, 2009).

Submerged macrophytes are plants which grow entirely underwater. Most of the submergent aquatic plants are lack of roots. The stems and leaves of this species are soft in nature and the leaves are mostly highly divided so that the plants are flexible enough to withstand water movement without damage (Cronk & Fennesy, 2009). The study done by Crowder *et al.* which cited in Hassan *et al.* (2009) showed that submerged species accumulate much more metal in their tissue than floating and emergent species.

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Free floating plants refer to the species that rooted under the water with floating leaves which constitute the most part of the plant mass. The floating plants move on water surface with winds and water currents. The roots absorb nutrient and help to stabilize the plant on water. The mineral supply for this species comes from water and partly from atmosphere as they are not touching the bottom of sediment (Heisey & Damman, 1982). Examples of floating aquatic macrophytes are duckweed, water hyacinth, water lettuce and water lily.

The roots of emergent plants are beneath the water surface whereas the leaves, stems and flowers grow out of the water surface. Emergent species are the most similar to terrestrial plants among all aquatic macrophytes (Cronk & Fennesy, 2009). They usually found in the boundary between dry land or wetlands and the open water littoral zone of lakes.

1.1 Research Objectives

The objectives of this research are:

i.   to investigate the influence of initial concentration and contact time on the adsorption of Cu (II) and Pb (II) ions by using dried water lily leaves;
ii. to study the adsorption isotherm on dried water lily leaves; and
iii. to examine the kinetic mechanism of the Cu (II) and Lead (II) ions adsorption.

CHAPTER 2

LITERATURE REVIEW

2.1 Copper

Copper is a transition metal that located in Group 11 and Period 4 of the periodic table. Its atomic mass is relatively 63.55 g/mol. The boiling point and melting point of copper is 2595 °C and 1083 °C, respectively. It exists in solid form at room temperature. It is used as thermal conductor and electrical conductor due to its high thermal and electrical conductivity. There are four oxidation states of copper which include +1, +2, +3 and +4. Usually, copper appear as Cu^{2+} which is blue or green in colour. The ductile and flexible properties of copper enable it to be drawn into wires. Copper is essential to human life and health. However, like all heavy metal, it is potentially toxic (Aydin *et al.*, 2007).

2.1.1 Sources of Copper

Copper is very common to be found in natural environment. For instance, native copper occurs abundantly as chemically uncombined mineral. The sources of minerals include sulphides such as chalcopyrite ($CuFeS_2$), bornite (Cu_5FeS_4), covellite (CuS) and chalcocite (Cu_2S). Other than that, the minerals of copper also exist in oxides form which is cuprite (Cu_2O). Copper can be released into the environment through wind-blown dust, decaying vegetation, forest fires and sea spray. According

to Rao *et al.* (2005), copper sulphate is used widely as an algicide in ornamental ponds which facing alga-bloom problems.

Furthermore, copper is introduced into the environment by anthropogenic activities. Mining, metal production, wood production, waste disposal, landfills and industrial wastewater are the examples of activities which contribute to the release of copper. In addition, copper bearing mining wastes and acid mine drainage discharge significant quantities of dissolved copper into wastewater (Rahman & Islam, 2009). Benaissa & Elouchdi (2006) also stated that meal cleaning, plating baths, fertilizer industry and municipal water runoff are the potential sources of copper in industrial effluents.

2.1.2 Pollution of Copper

There are many actual and potential sources of pollution from copper as it is a widely used material (Yu *et al.*, 2000). Copper may be found as contaminant in food especially shellfish, mushrooms, nuts and chocolate. The pollution of copper in wastewater arises from brass manufacture, electroplating industries and excessive usage of Cu-based agro-chemicals. It is recognized as one of the metals which are the highest relative mammalian toxic. Hence, the maximum acceptable level of copper in drinking water is less than 3000 $\mu g/dm^3$ (Rao *et al.*, 2005).

According to Dermirbas *et al.* (2007), most metals from the fourth period of periodic table are carcinogenic due to their electronic transition structure and inner transitional metals. The most toxic form of copper is Cu^{2+}. Furthermore, copper is phytotoxic at relatively low concentrations. It is a trace metal that required by plants for survival (Cardwell *et al.*, 2002). Nonetheless, it will reduce photosynthetic activities in plant when in excessive levels and hence, plant growth decreases (Wang *et al*, 1997). Copper's toxicity is highly pH dependent. It is found to be more toxic to living organisms at lower pH values.

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In human body, the excess copper accumulates in liver. It causes severe mucosal irritation and corrosion, capillary damage, hepatic and renal damage, severe gastrointestinal irritation and possible neurotic changes in liver and kidney (Benaissa & Elouchdi, 2006). Furthermore, copper acts as an irritant to the skin causing itching and dermatitis which will then lead to keratinisation of hands and soles of the feet (Rahman & Islam, 2009). Moreover, continued inhalation of copper-containing sprays will result in higher exposure to lung cancer (Ozcimen & Ersoy-Mericboyu, 2009).

2.1.3 Removal of Copper

There is a long history of human exposure to abnormal high levels of toxic metals n food and drink resulted from practices such as cooking in copper-lined pots and the water supply through copper pipes. Furthermore, there are industrial wastes disposal of metals into water bodies (Demirbas *et al.*, 2007). Thus, removal of toxic metals such as copper is extremely important. According to Eckenfekder Jr. (2000), copper is removed from wastewaters by precipitation or recovery processes such as ion exchange and adsorption.

Agricultural wastes have been used as sorbent for the removal of metal ions due to their broad availability and low cost. In the study done by Benaissa & Elouchdi (2006), the sorption capacity of dried sunflower leaves was investigated along with several parameters such as initial metal ions concentration, contact time, and initial pH of solution. The results showed that sorption of copper increased as contact time, initial metal concentration and initial pH of solution increased. This phenomenon is because of the increase in driving force of the concentration gradient. The maximum sorption of copper was found to occur around pH 5 – 6. Next, the required time to reach equilibrium was found to be initial copper ions dependent. The range for necessary contact time is 2.5 – 7 h.

The adsorption capacity for Cu (II) and Pb (II) of powdered activated carbon prepared from *Eucalyptus camaldulensis Dehn.* bark was tested. The effects of pH, contact time, initial metal concentration and temperature were of interest in the study. Results showed that the best adsorption occurred at pH 5, where the equilibrium time is found to be within 45 min for the range of initial metal concentration (0.1 – 10 mmol/L). The maximum adsorption capacity (q_e) for Cu (II) and Pb (II) occurred at 60 °C with the values of q_e of 0.85 and 0.89 mmol/g, respectively (Phussadee *et al.*, 2008).

There is growing interest in using low–cost materials for the adsorption of heavy metal. The potential of coir as a low–cost adsorbent for lead and copper is assessed. The experiment examined influence of pH, contact time, sorbate concentration, particle size and sorbent mass on adsorption. The researchers found that pH was a crucial factor in determining the adsorption for both metals. Other than that, lead had been proven to have a higher sorption capacity than copper which is 48.8 and 19.3 mg/g, respectively. Lastly, the kinetic study showed that the adsorption rates were a function of initial sorbate concentration, the particle size and mass of sorbent being used (Quek *et al.*, 1998).

Removal of Cu (II), Pb (II) and Cd (II) from aqueous solution by using *Ulmus carpinifolia* and *Fraxinus excelsior* tree leaves were evaluated. In this experiment, the effect of pH, initial metal concentrations, and contact time were studied. Results proved that the maximum sorption for *U. carpinifolia* and *F. excelsior* tree leaves for Cu (II) are 69.5, 33.1 mg/g, Pb (II) are 201.1, 172.0 mg/g and Cd (II) are 80.0, 67.2 mg/g, respectively. The most effective range for pH is between 4 – 5 for Cu (II), 2 – 5 for Pb (II) and 3 – 5 for Cd (II). The biosorption capacities for metal ions were in following order: Pb (II) > Cd (II) > Cu (II). The results from this study showed a novel way of using *U. carpinifolia* and *F. excelsior* tree leaves to remove Cu (II), Pb (II) and Cd (II) from metal polluted waters (Sangi *et al.*, 2007).

Sawdust, which is a by-product of world industry, was found to be a low cost and promising adsorbent for removal of Cu (II) from wastewater. The adsorption of copper was investigated by applying batch techniques in the study. Solution pH, contact time and sorbate concentration were the factors which affect equilibrium adsorption level. The maximum removal is achieved about pH 7 with the adsorption capacity of 1.79 mg/g. Next, the isothermal data of copper sorption are fitted into both Freundlich and Langmuir isotherms (Yu *et al.*, 2000).

2.2 Lead

Lead is a soft and malleable metal with atomic number 82. It belongs to Period 6 and Group 14 in the periodic table. At room temperature, lead appears as solid with bluish white colour. It boils and melts at 1749 °C and 328 °C, respectively. It is found abundantly in the earth's crust. The electrical conductivity of lead is relatively low. Lead is very resistant to corrosion but tarnishes when exposed to air. It is a common industrial metal that widespread in air, water, soil and food (Yu *et al.*, 2001). Lead is odourless, tasteless and colourless in solution (Gray, 2002).

2.2.1 Sources of Lead

Metallic lead is rarely occurs in nature. It usually found in ore with zinc, silver and copper. Lead is mostly found in galena and some found in pyromorphite, boulangerite and cerussite. It may be exposed to the environment through lead-containing paint, pluming leachate, ceramic ware and leaded petrol. Other than that, lead particles are emitted from mines and smelters in the form of $PbSO_4$, PbO. $PbSO_4$, and PbS. The main sources of lead in water are the effluents of processing industries (Ozgul & H. Ferdi, 2007). In water, lead has a tendency to form compounds of low solubility with major anions. They may present in the form of lead carbonate, lead oxide and lead hydroxide.

All lead compounds are considered as cumulative poisons (Imamoglu & Tekir, 2007). The toxic effect of lead is shown even at relatively low concentration. According to Salim & El-Halawa (2002), lead, along with cadmium, is probably considered as the most hazardous metals. Their toxicity is especially dangerous due to their cumulative nature. Acute lead poisoning can lead to gastrointestinal and nervous system failure. The health effects that caused by exposure to lead include irreversible brain damage and injury to blood forming system. Furthermore, the metal can cross the placenta and damage the developing fetal nervous system (Yu *et al.*, 2001).

2.2.2 Removal of Lead

In order to remove Pb (II) ions from aqueous solution, several studies have been done. The sorption of Pb (II) ions onto peat was studied by using batch technique. The investigation was carried out to study the effect of initial concentration, agitation and temperature on the adsorption capacity. The result showed that the significant sorption capacity for Pb (II) ions by peat is 122 mg/g. The effect of agitation rate is not significant on the time needed to reach equilibrium. The quantities of Pb (II) ions adsorbed onto peat increased when the initial metal ions concentration increased. The equilibrium sorption isotherm is fitted into Redlich-Peterson and Langmuir isotherms with correlation coefficients of 1.000 and 0.999, respectively (Ho & McKay, 1999).

Removal of Pb (II) and Cu (II) ions from aqueous solutions were carried out by using raw pomegranate peel. The batch experiments were performed as a function of pH, contact time, solute concentration and adsorbent dose. The study showed that optimum pH for maximum adsorption was found to be 5.6 and 5.8 for lead and copper, respectively. Other than that, the rate of removal is found to be higher in the beginning due to large surface area of adsorbent. Lastly, the percentage of removal for metal ions decreases as the initial concentration of solution increases (El-Ashtoukhy *et al.*, 2007).

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