MAGNETIC WASTEWATER TREATMENT SYSTEM BY USING MAGNETIC POLYAMIDE EPICHLOROHYDRIN AS ADSORBENTS

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A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering Technology

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> > NOVEMBER 2006

Declaration Page

I declare that this thesis entitled "Magnetic wastewater treatment system by using magnetic polyamide epichlorohydrin as adsorption medium" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Dedication

To my lovely mother for her love and encouragement, To my beloved and respected father, To my HU-HA siblings, &

To my only ONE for her existences and support -Adham Daslina-

Acknowledgement

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ABSTRACT

The main objectives of this study are to reduce concentration of lead in wastewater and to fabricate a new system based on magnetic and adsorption concept. In this study, prototype of wastewater system is fabricated. This prototype used a hardware components and electrical components. The system completed with existences of adsorbent. The magnetic polyamide epichlorohydrin (MPE) is used as adsorbents and it is produced by reaction of major chemicals; epichlorohydrin, ammonia solution, polyethlenemine and activated ferrite. This adsorbents assumed could reduced the lead concentration in wastewater. The parameter in this study is duration of cycle. A result conducted base on 5 min of cycle, 10 min of cycle, 15 min of cycle, 20 min of cycle, 25 min of cycle and 30 min of cycle. Based on the result of experiment, the concentration of lead reduced about 82% of the concentration before. This experiment concludes that magnetic polyamide epichlorohydrin is suitable to reduce lead.

ABSTRAK

Penyelidikan ini bertujuan untuk mengurangkan kandungan kepekatan plumbum dalam sisa air buangan. Dalam penyelidikan ini, satu sistem penapisan telah direka berdasarkan konsep magnet dan penjerapan. Sistem baru ini menggunakan komponen hardware dan juga komponen elektrik. Sistem ini lengkap dengan wujudnya resins. Resins yg digunakan adalah magnetic polyamide epichlorohydrin(MPE). Resins ini dihasilkan melalui tindakbalas beberapa bahan kimia iaitu epichlorohydrin, larutan ammonia, polyethlenemine dan serbuk besi. Resins ini dikatakan dapat mengurangkan kepekatan kandungan plumbum dalam air sisa buangan. Parameter yang digunakan dalam penyelidikan ini ialah masa kitaran untuk 5min,10min, 15min,20min,25min dan 30min. Berdasarkan keputusan eksperimen yang dijalankan, kepekatan kandungan plumbum berkurangan diantara 82% berbanding kepekatan sebelum. Daripada eksperimen, dapat disimpulkan bahawa magnetic polyamide epichlorohydrin sesuai untuk mengurangkan plumbum.

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NOMENCLATURE

- **B** Magnetic field, in (teslas).
- μ_o Permeability constant (1.26x10⁻⁶ H/m)
- i Current in the wire, in amperes.
- **N** Total number of turns of wire in the toroid

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CHAPTER I

INTRODUCTION

1.1 Introduction

Wastewater is sewage, storm water and water that been used for various purposes around the community. Unless properly treated, wastewater can harm public health and the environment. Most communities generate wastewater from both residential and nonresidential sources. Generally wastewater could be treated by doing 4 steps of treatment which are preliminary treatment, primary treatment, secondary treatment and final treatment [1].

Substances that are removed during the process of water treatment include an inorganic matter such as lead. A lot of methods could be used to treat lead. One of them is magnetic wastewater treatment. This method is agreed could reduce actinide and heavy metals particles in wastewater.

Magnetic Water Treatment is kindly about magnetic and the resins that work together to filter the heavy metals. It precisely use principles of magnetic to force the heavy metals permeate from contaminated water and some additional principles of adsorption that adsorb the particles in waste water.

The adsorption mechanism of ferrite occurs through metal hydroxide species. In alkaline solution, most metal ions form insoluble species which bonding with additional hydroxide ions, depending upon the pH of the solution and the chemistry of the metal. Hydroxide ions are strongly adsorbed onto the surface of ferrite

particles, so that metal ions can bond to the ferrite surface through the hydroxide ions to which they are bound. This mechanism is consistent with the phenomenon that alkali, alkaline earth and other metals, which do not form insoluble hydroxides, are not removed from solution by ferrites. Metals which form soluble hydroxide complexes at high pH, such as aluminum and zinc, also are not affected by ferrite. Beside that, it is known that ferrite can be applied to waste water treatment in various physical forms. Natural magnetite needs activation to have same capacity as freshly prepared ferrite. Magnetite ore can be effectively activated by various treatments to increase its surface area and adsorptive characteristics. There is being synergetic effect in using supported ferrite using column mode with an external magnetite field for concentration of heavy metals from waste water. This system will be prepared by attach the coils to the outer of resin bed while the beds interstices between the bed that promote good flow through properties [4].

According to the standard B water quality 1979, the concentration of lead allowed to be dump into river is 0.5 ml/g [5]. Therefore, the focus of this study is to reduce the concentration of lead by using magnetic wastewater treatment system.

1.2 PROBLEM STATEMENT

Lead that contain in wastewater could affect human life and also aquatic population. Lead gives a big impact to our health. Untreated wastewater that contained lead could harm human health. It persistence give a lot of consequences if this undesired heavy metals consistency located in blood. Lead induced toxicity to the central nervous system has been found to cause delayed development, diminished intelligence, and altered behavior. Some studies suggest that there may be a loss of up to 2 IQ points for a rise in blood lead levels from 10 to $20\mu g/dl$ in young children [2].

1.3 OBJECTIVES OF STUDY

To solve the problems above, two objectives have been made:

- 1) To reduce concentration of lead in wastewater.
- 2) To fabricate a new system based on magnetic and adsorption concept.

1.4 SCOPE OF STUDY

To achieve the objectives above, the scopes have been made:

- 1) Design and fabricate adsorbents system by using magnetic polyamide epichlorohydrin as adsorbents.
- 2) Effect of different duration of recycling process on lead concentration.

CHAPTER II

LITERATURE REVIEW

2.1 Wastewater

Wastewater is effluent from homes, commercial establishments, industries, public institutions, and similar entities have used their waters for various purposes. It is synonymous with sewage, although sewage is more general term that refers to any polluted water, which may contain organic and inorganic substances, industrial wastes, groundwater that happens to infiltrate and to mix with the contaminated water. The wastewater components that should be of most concern to homeowners and communities are those that have the potential to cause disease or detrimental environmental effects.

2.1.1 Wastewater Contents

Table 2.1 shows the list of wastewater contents such as organism, pathogens, organics and inorganics, nutrients, oils and greases, solids, gases, and household hazardous wastes.

Table 2.1: Wastewater Contents

Residues	Description
Organisms	Bacteria, protozoa, and worms
Pathogens	Viruses, parasites, and bacteria
Organics	Paper products, detergents, cosmetics, foods, agricultural,
	commercial, and industrial sources.
Inorganics	Sodium, potassium, calcium, magnesium, cadmium,
	copper, lead, nickel, and zinc.
Oils and greases	Fatty organic materials from animals, vegetables, and
	petroleum.
Nutrients	Nitrogen and phosphorus in the form of nitrate and
	phosphate.
Solids	Settleable solids, suspended solids and dissolved solids.
Gases	Ammonia, hydrogen sulfide, methane
Household	Transmission fluid, antifreeze, paint, paint thinner, varnish,
hazardous wastes	polish, wax, solvents, pesticides, rat poison, oven cleaner,
	and battery fluid, motor oils.

2.1.2 Wastewater Treatment Methods

There are many steps which are recommended for wastewater treatment steps. As usually there are 4 general steps which are preliminary treatment, primary treatment, secondary treatment and final treatment. Sometimes, advanced treatment will be use on specific purpose. All the details of the method are explained in table 2.2



Figure 2.1: General treatment step (source: http://ohioline.osu.edu/aex-fact/0768.html)

Based on the Figure 2.1 above, there are four steps of water treatment. Preliminary treatment consist two types of methods which are screening and storage. Primary treatment includes coagulation, flocculation and sedimentation. For Secondary treatment, there are 4 methods which are filtration, disinfection, distillation and reverse osmosis. Lastly for final treatment, methods like ion exchange and Electrodeionization. Table 2.2 below explained all about the treatment methods.

Table 2.2 Shows the treatment methods

Treatment	Treatment	Description
Classification	Method	
		The first step in purifying surface water is to
Screening	Screening	remove large debris like sticks, leaves, trash
	and other large particles which may interfere	

Preliminary	with subsequent purification steps		
		Water from rivers may also be stored in bank	
		side reservoirs for periods between a few	
	Storage	days and many months to allow natural	
		biological purification to take place.	
		Many of the suspended water particles have a	
		negative electrical charge. The charge keeps	
		particles suspended because they repel similar	
	Coagulation	particles. Coagulation processing reduces the	
		surface charge to encourage attraction which	
		forms floc which can settle.	
		Flocculation is the clumping together of small	
		particles to form larger particles, called floc,	
Primary		which is more readily settled out of the water.	
	Flocculation	Flocculation is the main method to decrease	
		turbidity. After charge neutralization of	
		suspended particles, they will stick to each	
		other and to the coagulant chemical particles.	
	Sedimentation	Water exiting the flocculation basin enters the	
		sedimentation basin, also called a clarifier. It	
		is a large tank with slow flow, allowing floc	
		to settle to the bottom.	
	Filtration	After separating most floc, the water is	
		filtered as the final step to remove remaining	
		suspended particles and unsettled floc. The	
Secondary		top layer removes organic compounds which	
		could include dangerous disinfection by-	
		products as well as those with taste and odor.	
		Disinfection with aggressive chemicals like	
	Disinfection	chlorine or ozone is normally the last step in	
		purifying drinking water. Water is disinfected	

	1	
to destroy any pathe		to destroy any pathogens which passed
		through the filters.
		Distillation involves boiling the water to
	Distillation	produce water vapor. The vapor contacts a
		cool surface where it condenses as a liquid.
	Reverse osmosis	Mechanical pressure is applied to an impure
		solution to force pure water through a semi-
		permeable membrane. Reverse osmosis is
		theoretically the most thorough method of
		large scale water purification available,
		although perfect semi-permeable membranes
		are difficult to create.
Final	Ion exchange	. A more rigorous type of ion exchange swaps
		H+ ions for unwanted cations and hydroxide
		(OH-) ions for unwanted anions. This system
		is recharged with hydrochloric acid and
		sodium hydroxide, respectively. The result is
		essentially deionized water.
		Ion selective membranes allow the positive
	Electrodeionization	ions to separate from the water toward the
		negative electrode and the negative ions
		toward the positive electrode.

2.2 Heavy Metals

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

2.2.1 Lead

Lead maybe released into the general environmental as a result of automobiles using gasoline as an antiknock agent, lead containing paint, water due to lead solder in water pipe systems, and atmosphere emissions of lead for industrial sources such as smelters. In recent years, that atmospheric release of lead has been reduced in many countries owing to the removal of lead from gasoline and restrictions on the release of lead from point sources. Lead based paint is still a problem in some countries such as United States, as a result of its use in older housing stocks. The decreased use of lead solder in water systems has greatly reduced exposure to lead from this source. In humans exposure to lead can result in a wide range of biological effects depending on the level and duration of exposure.

Various effects occur over a broad range of doses, with the developing fetus and infant being more sensitive than the adult. High levels of exposure may result in toxic biochemical effects in humans which in turn cause problems in the synthesis of hemoglobin, effects on the kidneys, gastrointestinal tract, joints and reproductive system, and acute or chronic damage to the nervous system.

Lead poisoning, which is so severe as to cause evident illness, is now very rare indeed. At intermediate concentrations, however, there is persuasive evidence that lead can have small, subtle, sub clinical effects, particularly on neuropsychological developments in children.

2.2.2 Physical properties of lead

Pure lead has little strength and is very soft and malleable. Because its interatomic bonding forces are weak and its melting point is very low, any stresses applied to pure lead may be relieved at normal ambient temperatures by recrystalisation and/or grain growth with a resulting permanent deformation of the lead. Table 2.3 below shows lead properties.

Table 2.3 Shows Lead properties

Properties	Values
Atomic mass (weight) in atomic mass units or grams	207.21
Atomic number	82
Melting point	327.4°C
Boiling point	1,750°C
Density (g/cm3)	11.34

Coefficient of linear thermal expansion	29.3 x 10-6/K
Specific Heat	129.8 J/kg K

2.2.3 Chemical properties of lead

Perhaps lead's best known property is its resistance to corrosion in various aggressive environments especially sulphuric acid. Lead's ability to give good service in such situations often gives the erroneous impression that lead is a passive metal. Lead is, in fact, a very reactive metal and it is this reactivity which enables it to be used in corrosive environments. In air, for example, a close fitting and adherent film of lead carbonate is formed by rapid reaction first between metallic lead and oxygen to form lead oxide followed by a second reaction between the lead oxide film and carbon dioxide, which is always present in air, to form a protective film of lead carbonate. Further contact with the metallic lead underneath is then prevented and corrosion ceases.

2.3. Epichlorohydrin

Epichlorohydrin is a chemical intermediate used primarily in the manufacture of epoxy resins and synthetic glycerol. It is also used in the production of epichlorohydrin elastomers, polyamide-epichlorohydrin resins, water treatment chemicals, and a variety of glycidyl derivatives. Figure 2.2 showed the formula for epichlorohydrin. Meanwhile table 2.4 showed the characteristic of epichlorohydrin that useful to be used in producing adsorbents.



Figure 2.2 Formula of chemical substance, Epichlorohydrin (Source: http://www.tocc.co.th/products.htm)

Table 2.4 Characteristics	of epichlorohydrin
---------------------------	--------------------

Items	Properties
Appearance	Clear colorless liquid
Odor	Irritation odor similar to chloroform
Molecular weight	92.53
Specific gravity	1.18 (20/4 OC)
Viscosity	1.03 cp. (28 0C)
Melting point	-25.6 0C
Boiling point	117.9 OC
Vapor pressure	1.6 Kpa (12 mm Hg) (20 0C)
Vapor density	3.19 (Air =1)
Solubility in water	6.6 Wt% (20 0C)
Refractive Index	1.43585 (nD25)
Surface tension	37.0 dynes/cm (20 0C)
Electrical Conductivity	20.8 (21.5 OC 1 = 60 cm)
Dielectric constant	97.9 cal/g

2.4 Ferrite

Ferrite is a class of ceramic material with useful electromagnetic properties and an interesting. Ferrite is rigid and brittle. Like other ceramics, ferrite can chip and break if handled roughly. Luckily it is not as fragile as porcelain and often such chips and cracks will be merely cosmetic.

Ferrite varies from silver gray to black in color. The electromagnetic properties of ferrite materials can be affected by operating conditions such as temperature, pressure, field strength, frequency and time. There are basically two varieties of ferrite: soft and hard. This is not a tactile quality but rather a magnetic characteristic. 'Soft ferrite' does not retain significant magnetization whereas 'hard ferrite' magnetization is considered permanent. Ferrite has a cubic crystalline structure with the chemical formula MO'Fe₂O₃ where Fe_2O_3 is iron oxide and MO refers to a combination of two or more divalent metal (such as : zinc, nickel, manganese and copper) oxides (<u>www.fair-rite.com</u>, 1 February 2006). Figure 2.3 showed illustrated of ferrite powder.



Figure 2.3 Ferrite powder (Source: http://www.e-magnet.cn/productsh1.html)

2.5 Solenoids

A solenoid is a long, tightly wound coil carrying electric current. The magnetic field generated by the solenoid is very strong inside the coil. Outside of the solenoid the magnetic field is essentially zero. Figure 2.4 showed the illustrated solenoid concept that produce magnetic field



Figure 2.4 Solenoid to produce magnetic field (Source: http://hyperphysics.phy-astr.gsu.edu/hbase/magnetic/solenoid.html) 2.5.1 Field inside an air core toroid coil

Figure 2.5 showed the illustrated air core toroid solenoid which produced magnetic field inside the column. The formula below showed the calculation of magnetic field strength



Figure 2.5 Air core toroid solenoid.

(Source: http://www.netdenizen.com/emagnet/solenoids/frommaxwellonly.htm)

$$B = \frac{\mu_o i N}{2\pi r} \tag{1}$$

Whereby:

B is the magnetic field, in teslas.

 μ_{o} is the permeability constant (1.26x10⁻⁶ H/m)

i is the current in the wire, in amperes.

N is the total number of turns of wire in the toroid

2.4 Fundamental of Magnetic Adsorption

Adsorption can be defined as a process leading to a significant increase in particle concentration occurring in a thin layer adjacent to an interface. The driving forces of adsorption are the short-range interactions between particles and interfaces, often called colloid or surface interactions. According to this definition, a physical accumulation of particles caused, such as by gravity (sedimentation) is not classified as adsorption. However, adsorption viewed as a form of partitioning is a general notion comprising phenomena such as deposition, defined as irreversible adsorption, or adhesion when a chemical-type contact between a particle and an interface is created. Effective adsorption of colloid and bioparticles is important for many practical processes such as water and waste water filtration, papermaking, xerography, protein and cell separation, immobilization of enzymes, etc.

Adsorption is the binding of molecules or particles to a surface, must be distinguished from absorption, the filling of pores in a solid. The binding to the surface is usually weak and reversible. Just about anything including the fluid that dissolves or suspends the material of interest is bound, but compounds with color and those that have taste or odor tend to bind strongly.

It has been demonstrated that mixed valence iron oxide compounds, or ferrites, are effective in removing heavy metals and suspended solids from aqueous media. Due to their unique chemical structure, ferrites may contain one or more iron atoms capable of being replaced by other metal ions. This characteristic, together with ferrites' strongly magnetic character, make them particularly suited to applications associated with waste water decontamination

`Ferrites have also been demonstrated to be effective in removal of actinides (thorium, uranium, plutonium and americium) from waste water generated at the nation's nuclear weapons production facilities. Ferrite removal of actinides can be accomplished using several different techniques including mixing prepared ferrites with aqueous wastes and preparing ferrites in situ in waste solutions.

A different approach to waste water decontamination involves the use of anion exchange resins for the removal of various contaminants from aqueous media. Polyamine-epichlorohydrin-type resins were first demonstrated to be effective weakly basic anion exchange resins approximately thirty years ago. C. A. Feldt and G. T. Kekish, "Weakly Basic Anion Exchange Resins," U.S. Pat. No. 3,092,617 (1963). Organic ion exchange resins are commonly used in chemical treatment of contaminated water in both industrial waste water treatment and general water resources engineering. Extensive research efforts have been made to develop organic ion exchange resins that are suited to adsorbing particular organic and inorganic contaminants. Alone, however, organic ion exchange resins are not especially effective for purposes of removing actinides from water, especially since noncharged polymeric species can be present in the water.

Due to the fact that large mounts of waste water containing heavy metals, or actinides, or both, are being generated, stored and disposed of in the United States and elsewhere, improved methods for handling and decontaminating such waste water are needed. The present invention helps to serve this need by providing a highly effective means for removing heavy metals and actinides from water.

The described invention greatly enhances removal of actinide and heavy metal contaminants from water as compared with existing water decontamination processes. By joining magnetic ferrites with polymer resins to produce magnetic ion exchange resins, and by utilizing the resins in the presence of a magnetic field, the invention yields markedly improved decontamination results due to an apparent and unexpected synergism between the individual mechanisms associated with ferrite decontamination systems; ion exchange resins and an external magnetic field.

Accordingly, it is an object of the present invention to provide a method for decontamination of wastes containing water and heavy metals (including, but not limited to cobalt, copper, arsenic, chromium, silver, lead, mercury, and cadmium, and combinations of those metals), beryllium, or actinides, or a combination of them, which utilizes magnetic ferrites, anion exchange resins, and a magnetic field in combinations.[9,10]

CHAPTER III

METHODOLOGY

3.1 Introduction

The methodology of the research is discussed in this chapter. The methods that are used in this research are fabrication and research. Fabrication is done first with selecting the suitable size of pipes. It is important to make sure the system could load maximum pressure of waste flow. This study only focuses on removing lead in wastewater by using magnetic wastewater treatment system.

3.11 The experiment flowchart

Experiment already runs to determine the concentration of lead adsorbed. This experiment is to study the lead concentration based on duration of cycle. This experiment concentrates on duration of cycle. Current, pressure and flowrate is set to be constant. Set the pressure gauge to be 1 Bar. The current supply form DC supply is set to be 4A. Then flowrate of waste is determined. Firstly the waste sample is pour into feed tank that have capacity 20 L. Before run the system, make sure check all parts of system to avoid undesired trouble such as pipes leaking. After satisfactory with the safety of all part of system, the system is run. Make sure there no trouble with pump and valve. Data of experiment is recorded for 5min, 10min, 15min, 20min, 25min and 30min. Figure 3.1 shows the flow of the experiment. It starts from developing the MWT system until writing the thesis.



Figure 3.1 Experiment Flowchart

3.2 System Design

To design this system, all equipment like pump, valve, elbow 90°, pressure gauge, flowrate meter is connected with PVC pipes 20mm inner diameter. To make sure the pressure in the PVC pipes not overload, 2 pressure gauges are located before the inlet of adsorption column and the exit of adsorption column. Flowrate meter also are installed to determine the flowrate and make it constant to avoid system leak. To connect the end of PVC pipes, elbow 90° are used. This is also important to minimize the space of the system. Here, PVC glues are used to make sure no leaking around the pipes system. If there are leaks at the pipe joint, it could affect the concentration leads that are taken. Peristaltic pump are installed to make sure the sample could enter the adsorption column. Figure 3.2 showed basic design of the system.



Figure 3.2 Design of system.

3.2.1 Transparent PVC pipes

The transparent PVC pipes are use to be the housing of adsorbents bed, which is 2.5 inch (6.3cm). Figure 3.3 showed transparent PVC pipe that have used as adsorption column.



Figure 3.3 Transparent PVC pipes

3.2.2 PVC pipes

All of system parts will be joined by using PVC pipe with diameter size 20mm. Figure 3.4 showed the PVC pipe that used as connectors with another parts.



Figure 3.4 PVC pipes

3.2.3 Solid nylon.

Solid nylon used to be cover lid of adsorption column. This is important to make sure the resins not mixed with wastewater. Solid nylon will be a holder for the transparent PVC pipes. Figure 3.5 showed the solid nylon.



Figure 3.5 Solid Nylon

3.2.4 Copper coils

Electrical coils will be used as solenoid to produce magnetic field. Figure 3.6 showed copper coils for adsorption column indeed to produce magnetic field inside the column.



Figure 3.6 Copper coils

3.2.5 Pressure gauge

This system contain a pressure gauge that will be use to determine the pressure flow from the adsorption column. Figure 3.7 showed the pressure gauge that used in the system to control the pressure in the adsorption column.



Figure 3.7 Pressure Gauge

3.2.6 Valve

To fabricate this system, 2 valves will be used. a valves is use to control the water from enter the adsorption column and the other one is use for pressure control. Figure 3.8 showed the ball valve in controlled the flowrate of liquid and pressure of liquid in the system.



Figure 3.8 Ball valve

3.2.7 Tank

Feed tank 20 L will be use to store the wastewater. Figure 3.9 showed the feed tank that used in the system as a storage of the contaminated of water in the system.



.Figure 3.9 Feed Tank

3.2.8 Plywood

Plywood is used to support the tank and the MWT adsorption column. The size of the plywood is 120cm x 170cm. Figure 3.10 showed the system base that used the plywood to support the system design from collapsed.



Figure 3.10 Plywood