STUDY ON THE ADSORPTION OF ZINC FROM WASTE IPA BY GRANULAR ACTIVATED CARBON

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> > JANUARY 2012



ABSTRACT

The spent Isopropyl Alcohol (IPA) is a schedule waste from electrical and electronic manufacturing industries which is also abundance in our country. The IPA waste containing high concentration of electronic waste mainly comprises a few heavy metals which has the potential of purifying and reusing the waste by removing heavy metals from it. The aim of the study was to find optimum adsorption parameter for Zink removal from the spent IPA by utilizing the granular activated carbon (GAC). Investigation was carried out by studying the influence of the initial solution pH value ranging from 4 to 10, different temperature value ranging from 25 to 35°C and also agitation speed using incubator shaker that operated for 50,100 and 150rpm in the batch reactor. The adsorption capacity on the molar basis of heavy metal Zink (Zn) were able to be stripped from the IPA. The maximum and most significant of zinc removal percentage is 89.7% with is from 1.4621mg/L to 0.1506 mg/L at parameter of 30.8°C, 98rpm and pH4.The colour of waste samples were able to change from grey to colourless and strong odor of waste IPA became less at the end of the experiment.





ABSTRAK

Alkohol Isopropyl (IPA) yang telah diguna adalah sisa berjadual daripada industri pembuatan elektrik dan elektronik yang mana ianya sangat terabaikan di negara kita.Sisa IPA yang telah digunakan mengandungi beberapa logam berat berkepekatan tinggi yang mana mempunyai potensi untuk dibersihkan dan digunakan semula dengan menyinkir logam berat dari sisa IPA. Tujuan kajian adalah untuk mencari parameter optimum penjerapan bagi penyinkiran Zink dari IPA yang telah digunakan dengan menggunakan butiran karbon teraktif (GAC). Penyiasatan telah dijalankan dengan mengkaji pengaruh nilai pH dari 4 hingga 10, perbezaan nilai suhu dari 25 hingga 35 darjah celsius dan dan juga kelajuan menggoncang menggunakan penggoncang incubator yang beroperasi pada 50,100 dan 150 rpm dalam sekumpulan reactor.Keupayaan untuk menjerat berdasarkan molar logam berat dari sisa buangan IPA telah dianalisis denagn menggunakan Spektrometer Adsorpsi Atom (AAS). Logam berat, zink berjaya diasingkan dari IPA.Peratusan penyingkiran zink pada maksimum adalah sebanyak 89.7% daripada 1.4621 mg/L kepada 0.1506mg/L pada parameter 30 darjah celsius dan pH4.Warna sampel IPA dapat diubah dari kelabu ke warna jernih.Bau IPA juga dapat dikurangkan.



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LIST OF SYMBOLS

b	Langmuir parameter related to the energy of adsorption (l mg ⁻¹)
Ce	metal concentration in solution at equilibrium (mg l^{-1})
C_0	initial liquid phase concentration (mg l^{-1})
п	Freundlich isotherm constant related to adsorption intensity
Q	Langmuir parameter related to the capacity of adsorption $(mg g^{-1})$
q _e	amount of adsorbed metal on the moss at equilibrium (mg g^{-1})
$R_{ m L}$	dimensionless separation factor
R^2	correlation coefficient
V	volume of solution (l)
W	mass of adsorbent (g)



CHAPTER 1

INTRODUCTION

1.1 Background of Study.

Isopropyl Alcohol (IPA) shown in **Figure 1.1** is a colorless, flammable chemical compound with a strong odor widely used as a cleaner and solvent in industry. The largest use for IPA is as a solvent (Isopropanol Material Safety Data Sheet, 2003).

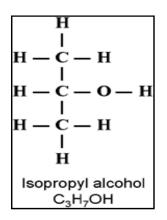


Figure 1.1: The molecular structure of IPA.

Source: David R. Lide ,1996.

In electric and electronic industries, IPA used for cleaning electronic devices such as, circuit board manufacturing (PCB), cleaning contact pins (like those on ROM cartridges), magnetic tape deck and floppy disk drive heads, as well as the lenses of



lasers in optical disc drives (e.g. CD, DVD) and removing thermal paste from CPU's waste (Alelstro, 2009).

IPA is produces by combining propene and water. There are two processes used in produce IPA, which are indirect and direct processes. During indirect process, hydrogen was involved and then hydrolysis of a petroleum product and propylene by using acid and water. The direct process involves hydrogenation of a petroleum product, propylene, with an acid catalyst (How, Azizan, Norfatyhah,Norasmanira and Izayu, 2010). Table below descript the physical properties of IPA.

Molecular Weight	60.09	
Boiling Point	82.5 °C	
Melting Point	-88.5 °C	
Flash Point	11.7 °C (53 °F) (closed cup)	
Vapor Pressure	44 mm Hg at 25 °C	
Density/Specific Gravity	$0.7851 \text{ at } 20/4 ^{\circ}\text{C} \text{ (water = 1)}$	
Log/Octanol Water Partition Coefficient	0.05	
Dissociation Constants	pka: 17.1 at 25 °C	
Autoignition Temp	455.6 °C (852 oF)	
Henry's Law Constant	8.07 x 10-6 atm-m3/mole	
Vapor Density	2.08 (air = 1)	
Conversion Factor	1 ppm = 2.45 mg/m3	

Table 1.1: Physical Properties of Isopropyl alcohol.

Source: HSDB, 1991.

Global IPA production capacity reached 2,153 thousand metric tons (4,747 million pounds) in 2003, although global capacity use was roughly 80%. Approximately 74% of the global IPA capacity is concentrated in the United States, Western Europe and Japan. Dow produced approximately 12% of the IPA in 2003 (Chemical Economics Handbook, 2003) at its site in Texas City, Texas, where it has 411 thousand metric tons (906 million pounds) capacity.

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In Alps Electric (Malaysia Sdn.Bhd), IPA is use as a cleaner agent for solder at PBC. Solder is use as a SMT or Surface Mount Technology. The solder is consists of composition heavy metals such as Bismuth (Bi), Stanum (Sn) and also Zinc (Zn).The spent IPA usually will be disposed at fill land as it is classify as a schedule waste which is cannot be saved for recovery or recycling by paying the third party contractor to handle the waste.

The absorption was chose as method for this experiment as it had been proven by Demirbas (2008) in his journal that, this method was "an excellent way to treat industrial waste effluents, offering significant advantages like the low-cost, availability, profitability, easy for operation and efficiency". In order hand," adsorption is considered a powerful technique that was extensively used for removal of heavy metal ions from domestic and industrial effluents" Bayramoğlu and Arıca, (2005).Beside, this method is choosing as it is suitable for low concentrations of metal ions. Adsorption models commonly chosen for comparison with experimental data are the Langmuir model, and the Freundlich model. There are two types of the adsorption which are physical adsorption and chemical adsorption or chemisorptions. The physical adsorption involves the van der Waal forces whereby the chemisorptions is about chemical bond between zinc and GAC (Akgün and Mert, 2005). The parameters such as capacity versus rate, surface area, pore size, particle size, and temperature, concentration of adsorbate, pH and contact time can be factors that affecting the adsorption process.

Activated carbon mainly use in the treatment of wastewater. It is used for purification, decolorization and the removal of toxic organics and heavy metal ions (Ain, 2007).Furthermore, the activated carbon is the best method proved by many researchers for removing heavy metals such as mercury (Huang and Blankkenship, 1984), copper (Netzer and Hughes, 1984), lead (Reed and Arunachalam, 1994), chromium (Park and Jung, 2001), cadmium (Rangel-Mendez and Streat, 2002), Ni (Shim, Park and S.-K. , 2001), zinc (Monser and Adhoum, 2002), and lithium (Rangel-Mendez and Streat, 2002).





Granular activated carbon has a larger particle size compared to powdered activated carbon but, presents a smaller external surface. Thus, diffusion of the adsorbate is an important factor.GAC can be either in the granular form or extruded (Geankoplis, 2003). Particle size: larger than 0.1 mm (100 mm). Typical range: 0.4-2.5 mm. According to New World Encyclopedia GAC is designated by sizes such as 8x20, 20x40, or 8x30 for liquid phase applications which is needed for this experiment.

The heavy metal concentration was determined by the use of atomic absorption spectrophotometer, determination of heavy metals was done by using its specific lamp for each metal and at a specific wavelength. According to Ayhan Demirbas, the Freundlich model (Freundlich and Hatfield, 1926) and Langmuir model (Langmuir, 1918) is among the frequent model use in literature review to describe the experimental data of adsorption isotherms applied in batch system.

1.2 Problem Statement.

In manufacturing and electronics industries, IPA is widely used as a cleaner and drying agent to remove all the dirt, impurities from finished goods. The used IPA from this process contain high concentrations of electronics waste mainly comprises few types of heavy metals (Claus Christ, 1999). In order to optimize the IPA consumptions and reducing the environmental hazards, the best strategy to explore is by researching on the potential of reusing the spent IPA by removing as much heavy metals such as possible.



1.3 Research Objectives.

The objectives of this project are:

- i. To remove heavy metal zinc from spent IPA by using adsorption method.
- ii. To observe the effect of PH, temperature and agitation speed of mixing on the efficiency of zinc adsorption.
- iii. To find the best parameters to adsorb zinc from spent IPA.

1.4 Scope of Research.

In order to achieve the objectives of this study, the result will be measured by manipulate the pH, temperature and also agitation speed of mixing of batch adsorption process by using GAC as adsorbent. The analysis of adsorption efficiency will be done by analyzing the heavy metal (Zinc) concentration in the cleaned IPA by using (Atomic absorption spectroscopy) AAS.

1.5 Significance of Study.

Considering the magnitude of electronic waste in Malaysia, this research is expected to be able to address economic and environmental issues concerning the IPA waste from electronic industries. From the former aspect, the company will be able to reduce the cost of disposing IPA waste. In other word, the companies can save their budget in waste treatment because they can reuse the IPA that recycled from IPA waste rather than the current practice of paying external contractor to handle the waste. In the other hand, the precious heavy metal removed from the IPA may be process further to be purified back for other usage.

Secondly, heavy metal contain in IPA waste possibly can be toxic and can dissolve in ground and then seep into water supply. This not only can causing human's health problem but also polluted the river, aquatics life and ecosystem. According to a journal written by Reyad Shawabkeh, Adnan Al-Harahsheh and Awni Al-Otoom "Unlike organic pollutants, heavy metals do not degrade into less harmful substances



and can accumulate in soil and water reservoirs, which leads to harmful effects on living species if they enter into the food chain(2004)". Zinc is an important element that required for human health, however too much of zinc can cause eminent health problems such as stomach cramps, skin irritations vomiting, nausea and anemia (Oyaro et al., 2007). This shows a serious threat of such waste to our environment thus need to be handle properly.

Spent IPA also is abundance in our country which can provide continuous supply to the opportunities to save environment and the opportunities to recover precious heavy metals where all this while had been dumped or incinerated without any effort to take advantage on the potential to retract back the precious metals.

1.6 Research Questions.

- i. What is the observation of adsorption result when PH, temperature and agitation speed of mixing are manipulated?
- ii. Are the heavy metal, zinc contained in IPA waste are be able to be removed?
- iii. How to absorb the heavy metal from IPA waste using AAS?
- iv. What the best parameters to adsorb zinc from spent IPA?

1.7 Definition of Key Terms.

-**Isopropyl Alcohol (IPA)** - IPA also known as 2-Propanol; sec-propyl alcohol; isopropanol; sec-propanol; dimethylcarbinol. A colorless transparent combustible liquid, have be like ethanol smell. Soluble with water, ethanol, ethyl ether, chloroform immiscible.

-Print circuit board manufacturing (PCB) - a thin plate on which chips and other electronic components are placed.(Webopedia)

-Adsorption- One or more components of gas and liquid stream adsorbs onto a solid surface. This process commonly uses to remove of organic compounds from water.



-Atomic absorption spectroscopy (AAS) - Atomic absorption methods measure the amount of energy (in the form of photons of light, and thus a change in the wavelength) absorbed by the sample. The wavelengths of light transmit by the samples are measured by a detector, and compares them to the wavelengths, which originally passed through the sample. A signal processor then integrates the changes in wavelength, which appear in the readout as peaks of energy absorption at discrete wavelengths.

1.8 Conclusion.

The adsorption of zinc from waste IPA is choosing as it is abundance in our country which can provide continuous supply, save the landfill from IPA waste able to reduce the cost of disposing IPA waste, the precious heavy metal removed from the IPA may be can process further to be purified back for other usage and lastly zinc can cause eminent health problems such as stomach cramps, skin irritations vomiting, nausea and anemia if they enter into the food chain as it is do not degrade into less harmful substances and can accumulate in soil and water reservoirs, which leads to harmful effects on living species if they enter into the food chain.





CHAPTER 2

LITERATURE REVIEW

2.1 Introduction.

There are a lot of commercial method applied in industry to remove or separate heavy metal such as neutralization, membrane filtration ion exchange, precipitation; solid phase extraction and adsorption in fluids and gases from spent IPA from electronic waste. Among these methods, the adsorption is the most popular method using by researcher. Chunmei Niu, Wenhui Wu, Zhu Wang, Shumin LI and Jianquan Wang (2007) claim that adsorption is recognized as an effective, efficient and economic method for water decontamination applications and for separation to analytical purpose. This chapter will detail describe about the spent IPA and electronic waste, adsorption processes, definition and types adsorbents, and also adsorption isotherm that will be used and heavy metal involved in this experiment.

2.2 Spent IPA from Electronic Waste.

As discussed in the previous chapter, the IPA is use as a cleaning agent in electronic manufacture of PCB, pins in ROM cartridges, floppy disk and magnetic tape deck as well as the laser lenses of optical disc drives in CD and DVD player. The raw metal used by the industry in manufacturing PCB is commonly copper and sometimes aluminum, nickel and other metals for board. In electroplating process, copper, nickel, and tin/lead have been used and chromium and copper used for etching process (Lobby and Kirsh, 1992).



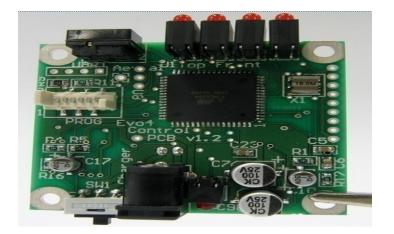


Figure 2.1: A PCB as a design on a computer.

Source: Michael, 2007.

According to Mr. Dzul Karnain B. Abd.Rahim, Senior Engineer from Alps Electric (Malaysia Sdn.Bhd), IPA is use as a cleaner agent for solder at PBC. Solder is use as a SMT or Surface Mount Technology which is involving either active (transistors, integrated circuits, diodes, and others.) or passive (capacitors, resistors, coils, and many more.) which do not have leaded connections. Terminal leads on the device are part of the component body, thus allowing direct mounting on the surface of printed circuit boards (PCBs). Leaded devices are mounted by passing interconnection leads through holes drilled and then plated in the PCBs.

The solder is consists of composition heavy metals such as Bismuth (Bi), Stanum (Sn) and also Zinc (Zn). There also Pb in spent IPA as the Alps Electronic Sdn. Bhd is also still use lead solder depending upon their customers demand. Thus, the heavy metals that may consist in the spent IPA from Alps Electric (Malaysia) Sdn. Bhd are Bismuth, Stanum and Zinc.

2.3 Adsorption.

The adsorption process can occur at an interface between any two phases such as liquid-liquid, gas-liquid, gas-solid or liquid-solid interfaces. It involves the interphase accumulation or concentration of substances at a surface or interface. The material being **Created with**



concentrated or adsorbed is the adsorbate and the adsorbing phase is called the adsorbent. When those adsorbed molecules are released from the interphase, desorption takes place (Weber., 1972; Akgün and Mert, 2005).

The absorption will be choose as method for this experiment as it had been proven by Demirbas (2008) in his journal that, this method was "an excellent way to treat industrial waste effluents, offering significant advantages like the low-cost, availability, profitability, easy for operation and efficiency".

In order hand," adsorption is considered a powerful technique that was extensively used for removal of heavy metal ions from domestic and industrial effluents" Bayramoğlu and Arıca, (2005).Beside, this method is choosing as it is suitable for low concentrations of metal ions. Adsorption models commonly chosen for comparison with experimental data are the Langmuir model, and the Freundlich model.

2.3.1 Types of Adsorption.

There are two types of the adsorption which are physical adsorption and chemical adsorption or chemisorption depending on the nature of the surface forces. The main different characteristic between these two types of adsorption is shown in table below:

Table 2.1: Summary of the characteristics of chemisorptions and physical adsorption.

Chemisorption	Physical Adsorption	
Releases high heat, 10 Kcal/gm mole	Releases low heat, 0.1 Kcal/gm mole	
Forms a chemical compound	Gas retained by dipolar interaction	
Desorption difficult	Desorption easy	
Adsorbate recovery impossible	Adsorbate recovery easy	

Source: Akgün and Mert, 2005.



2.3.1.1 Physical Adsorption.

In the physical adsorption, there are van der Waals forces between the adsorbed molecule and the surface of the adsorbent when the molecules are attached to the surface of the solid which are similar to those that cause vapor molecules to condense to liquid. It is a highly reversible process that required a low temperature, and is characterized by a relatively low energy of adsorption. In order to desorb the adsorbed material, vacuum or increase the temperature can be applied (Akgün and Mert, 2005).

2.3.1.2 Chemisorption.

Unlike physical adsorption, chemical adsorption or chemisorptions involves the formation of strong bonds between adsorbate and adsorbent surface. Chemisorptions is depends on the chemical nature of both the adsorbent and adsorbate. For example, the adsorption of oxygen on activated carbon. Because of the bonds between oxygen and carbon atoms are stronger than others atoms, increasing the temperature can be desorbs not as oxygen but as carbon monoxide or carbon dioxide (Akgün and Mert, 2005).

2.3.2 Factors Affecting Adsorption.

The parameters such as capacity versus rate, surface area, pore size, particle size, and temperature, concentration of adsorbate, pH and contact time that should be considered for this study can be referring from **Table 2.2**.





Parameters	Explanation		
Capacity vs.	a) Capacity parameters determine loading characteristics of		
Kinetics (Rate)	absorbent. Maximum adsorption capacity of absorbent is only		
	achieved at equilibrium.		
	b) Kinetic parameters only determine the rate of adsorption and		
	have negligible effect on adsorption capacity.		
Surface Area	Adsorption capacity is proportional to surface area (determined		
	by degree of activation).		
Pore Size	Correct pore size distribution is necessary to facilitate the		
	adsorption process by providing adsorption sites and the		
	appropriate channels to transport the adsorbate.		
Particle Size	Smaller particles provide quicker rates of adsorption. Note:		
	Total surface area is determined by degree of activation and		
	pore structure and not particle size		
Temperature	Lower temperatures increase adsorption capacity except in the		
	case of viscous liquids.		
Concentration of	Adsorption capacity is proportional to concentration of		
Adsorbate	adsorbate		
pН	Adsorption capacity increases under pH conditions, which		
	decrease the solubility of the adsorbate (normally lower pH).		
Contact Time	Sufficient contact time is required to reach adsorption		
	equilibrium and to maximize adsorption efficient.		

Table 2.2: The Factors Affecting Adsorption.

Source: Akgün, 2005.

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2.3.3 Adsorbent.

The well known and widely used adsorbent uses in variety application are activated carbon, activated Alumina, Zeolites and synthesis polymers (Vaughan, 1988). All of them have their specific characteristic for certain purpose. The table below descript the physical properties of major types of adsorbents.

Adsorbent	Internal Porosity (%)	Surface Area (m ² /gm)	Pore Volume (cm ³ /gm)	Bulk Dry Density (gm/cm ³)	Mean Pore Diameter (A)
Activated Carbon	55-75	600-1600	0.80-1.20	0.35-0.50	1500-2000
Activated Alumina	30-40	200-300	0.29-0.37	0.90-1.00	1800-2000
Zeolites (molecular Sieves)	40-55	600-700	0.27-0.38	0.80	300-900
Synthesis Polymers	-	1080-1100	0.94-1.16	0.34-0.40	-

Table 2.3: Physical Properties of Major Types of Adsorbents.

Source: Goltz, H.R.; Jones, K.C.; Tegen, and M.H., 1994.

2.3.3.1 Activated Carbon as Adsorbent.

Activated carbon mainly use in the treatment of wastewater. It is used for purification, decolorization and the removal of toxic organics and heavy metal ions (Ain, 2007).Furthermore, the activated carbon is the best method proved by many researchers for removing heavy metals such as mercury (Huang and Blankkenship, 1984), copper (Netzer and Hughes, 1984), lead (Reed and Arunachalam, 1994), chromium (Park and Jung, 2001), cadmium (Rangel-Mendez and Streat, 2002), Ni (Shim, Park and S.-K. , 2001), zinc (Monser and Adhoum, 2002), and lithium (Rangel-Mendez and Streat, 2002).



According to Steve Kvech and Erika Tuell in article titled Water Treatment Primer, they stated that, there are 3 basic steps activated carbon adsorption process which is:

- i. Substances adsorb to the exterior of the carbon granules
- ii. Substances move into the carbon pores
- iii. Substances adsorb to the interior walls of the carbon.

There are others types of activated carbon widely use in industry which is powder activated carbon. **Table 2.4** describes about the different between three type of activated carbon from their applications and sizes.

Table 2.4: The Different between Granular Activated Carbon (GAC), Powder Activated

 Carbon (PAC) and Activated Carbon Fibre.

Type of activated	Granular Activated	Powder Activated	Activated Carbon
carbon	Carbon	Carbon	Fibre
Applications	Purification of edible and inedible oils, purification of higher fatty acid, food industries(glucose, fructo se etc.),soft drink and effluent treatment,dye and dye intermediates, purification of acidic and basic electroplating baths.	Use for effluent and water treatment and different grades are identified by its lodine value. It is also used in the treatment of fumes and gases, with or without lime.	Air purification ,decolorisation,water purification, household product which is used in the refrigerator as a deodoriser.
Size	The size of particles is from 0.5 to 10 mm.	Size from between 1 and 100 µm.	Is synthetic material made from a poly acrylonitrile material which is activated at high temperatures for optimum activity.

Source: Mani and Sivakkumar, 2011 and Serpa, Schneider and Rubio, 2005.

