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SORPTION OF COPPER AND IRON USING POLY(HYDROXAMIC ACID) CHELATING RESIN.

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# THIS DISSERTATION IS WRITTEN TO FULFILL PART OF THE REQUIREMENT TO OBTAIN A BACHELOR DEGREE OF SCIENCE WITH HONOURS

PERPUSTAKAAN UNIVERSITI MALAYSIA SABAH

INDUSTRIAL CHEMISTRY SCHOOL OF SCIENCE AND TECHNOLOGY UNIVERSITI MALAYSIA SABAH KOTA KINABALU





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## DECLARATION

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#### ABSTRACT

This research is to analyze the poly(hydroxamic acid) resin as one of the polymer that can absorb metal ion especially ferum and cuprum. This research is to study the sorption capacity of metal ion especially iron and copper metal ion. Secondly is to research the rate of exchange capacity of the metal ion especially copper. The analysis is using the batch technique where each of the solution of polymer that contain about 0.2 gram was added with 20ml of vary acidity of buffer solution and water before the adding of vary ppm(part per million) of metal ion standard solution (Cu<sup>2+</sup> and Fe<sup>3+</sup> standard solution). The analysis of cuprum and ferum in the presence of poly(hydroxamic acid) chelating resin is to search the amount of each metal ion that can be absorb by resin or polymer in each gram of absorbance(polymer). Where the binding of the polymer with the metal ion is depend to the acidity of the solution used. From the analysis it's was found that the cuprum  $(Cu^{2+})$  is absorb by the polymer much better than the ferum (Fe<sup>3+</sup>). And at the high acidity condition the capacity of metal ion absorbs is much lower than the lower acidity. The highest number of absorb metal ion for ferum was 0.0525mg/g resin at the pH6. For cuprum metal ion the highest amount absorbs by polymer is also at the pH6 with the ppm5 that absorb about 0.1425mg/g resin. Totally the exchange capacity is increasing. And the capacity of metal ion absorb is depending on its solution acidity is increasing with the decreasing of acidity.



ABSTRAK

Tujuan kajian ini adalah untuk menunjukan poly(hydroxamic acid) resin boleh menjerap ion logam berat terutamanya kuprum dan ferum. Kajian ini juga adalah untuk menunjukan kapasiti jerapan logam ferum dan kuprum. Objektif yang kedua adalah untuk mengatahui kapasiti jerapan dengan masa pada ion kuprum oleh poly(hydroxamic asid) resin. 0.2g resin yang dicampurkan dengan 20ml air dan juga 20 ml larutan buffer pada keadaan pH yang berbeza dicampur dengan 10ml larutan logam. Bacaan diambil dengan AAS selepas 24 jam digoncang dengan shaker machine. Graf yang dilakarkan menunjukan bahawa pada keasidan yang tinggi resin tidak efektif menjerap logam dalam larutan. Apabila keasidan berkurang jerapan oleh polimer ini adalah tinggi. Polimer menjerap logam kuprum dengan lebih effisien berbanding dengan logam ferum. Bagi ferum jumlah tertinggi logam yang dijerap ialah 0.0525mg/g resin. Kuprum pula ialah 0.1425mg/g resin. Bagi kajian tentang kapasiti jerapan dengan masa menunjukan bahawa dalam jerapan akan meningkat dengan perubahan masa sehingga mencapai takat tertentu. Boleh disimpulkan bahawa keadaan berasid dan juga masa mempengaruhi jerapan oleh polimer ini.





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# SYMBOL LISTS

%	Percentage
А	Sorption
AAS	Atomic absorption spectrometer
cm	Centimeter
g	Gram
g/g	Gram per gram
mg/g	Milligram per gram
mmol/g	Milimol per gram
mg/L	Milligram per liter
ml	Milliliter
ppm	Part per million



# **OFFICIAL TERM LISTS**

Alkaline
Acid
Binding
Concentration
Copolymer
Cellulose
Complex ions
Chemical
Coordination complexes
Chelation
Determination
Equilibrium
Equilibration
Erlenmeyer flask
Extraction
Functional group
Free radical
Hydroxamic
Homopolymers
Heavy metals
Ligands

Beralkali Asid Ikatan Keseimbangan Copolymer Selulosa Ion kompleks Kimia Koordinasi complex Chelat Penentuan Keseimbangan Penyeimbangan Kelalang Erlenmeyer Pengekstrakan Kumpulan berfungsi Radikal bebas Hydroxamic Homopolimer Logam berat Ligand



Metal ion Methanolic solution Molecular weight Microelements Nitrile Oxidation Polymers Potassium cyanide Polymerization Redox Resin Selectivity Sorption

Transition metal

Ion logam Larutan metanolik Berat molekul Mikroelemen Nitril Pengoksidaan Polimer Natrium cynida Polimeran Redox Resin Redox Resin Pemilihan Serapan Larutan



# **CHAPTER 1**

#### INTRODUCTION

#### **1.1 INTRODUCTION**

A number of poly(Hydroxamic acid) resin have been synthesized by various method and for various purposes. Scientist have found that  $Fe^{3+}$ ,  $Cu^{2+}$ ,  $Ag^+$ ,  $Zn^+$ ,  $Hg^+$ ,  $Al^{3+}$ ,  $Pb^{2+}$  and  $TiO^{2+}$  were bound to the hydroxamic acid ligand. Gold is strongly bound to the resin and may only be quantitatively removed by use of potassium cyanide as eluant. In the present study the poly(Hydroxamic acid) chelating resin was used for separation of some transition metal ions from their binary mixture (Lutfor et, al., 2000).



Diagram 1.1: Poly(hydroxamic acid) resin



Pertains from the extraction of metals from fibrous homopolymers and copolymers of accrynitrile, divinylbenzena cross linked poly(acryloamidoxmine) resin were successfully applied to the determination of trace metals in natural water.

There are very few articles published on transition metal uptake by poly(amidoxamine) resin. This may first be introduced to the preparation of poly (amidoxamine) chelating resin from polyacrynitrile (PAN) grafted sago starch. In this study the PAN grafted copolymer was used as the cheapest starting material for poly(amidoxamine) resin preparation (Lutfor et, al., 2000).

The used of graft co-polymerization of methyl acrylate onto sago starch and other natural macromolecules can be initiated by transition metal ions was widely used in the modification of cellulose, fiber, starch etc. Redox system generally comprises transition metal ions which occur in various oxidation states. Such oxidized metal ions may directly oxidized starch and the free radicals are produced onto starch using ceric ion as an initiate the graft co-polymerization (Lutfor et, al., 2000).

Graft polymerization of monomers is one of the universal, effective, and accessible methods of chemical modification of high molecular weight compounds, natural polymers in particular. The advantage of radical polymerization is that it takes place in water media with good yield of the final products. Besides, it considerably widens the choices of monomers and the preparation of graft copolymers with wider ranges of Physico-chemical properties under technological process conditions. From the studies by Berlin and Kislenko about kinetics of radical graft polymerization of monomers onto polysaccharide, the rate of polymerization showed the first order



dependence on monomers concentration and a square root of initiator concentration (Lutfor et, al., 1999). The preparation of a chelating ion exchange resin containing amidoxamine functional group was carried out by polyacrylonitrile (PAN) sago starch. The PAN grafted copolymer obtained by free radical initiating process using ceric ammonium nitrate as an initiator. Conversion of nitrile groups of the grafted copolymers into the amidoxamine was carried out by treatment with hydroxylamine under alkaline solution (Lutfor et, al., 1999).

Scientists are exploring ways to use starch as a replacement for petroleum derived polymers in an effort to generate new markets for surplus starch and to increase the biodegradability of plastic articles. Combining starch with synthetic polymers through graft polymerization is one of the best approaches for achieving these goals. Graft polymerization of methyl acrylate onto starch gives materials that can be processed into tough and flexible plastic articles. The uses of grafted starch gives stronger materials with enhance water resistance. Grafting has been utilized as an important technique for modifying physical and chemical properties of polymers (Lutfor et, al., 2000).

Another approach consist of the introduction of amidoxime groups into cellose by the reaction of cyanoethylcellulose and acrylonitrile grafted cellulose with hydroxylamine pertains to the extraction of metals from dilute solution by poly(amidoxamine) derived from fibrous homopolymers and copolymers of acryloitrile. Divinylbenzene cross-linked poly(acryloamidoxamine) resin were successfully applied to the determination of trace metals in natural waters (Lutfor et, al., 2000).



### 1.2 Research objective

The main target here is to make the sorption of heavy metal ions using chalating resin. What we used here is we used the poly(hydroxamic acid) chelating resin that will be extracted from sago starch. And from the research tat bring by many scientist chelating resin s also can use in industry to separate metal ions from waste water. There are two main objectives in this study:

- 1. To study the sorption of copper and iron by poly(hydroxamic acid) chelating resin in the various pH.
- 2. To study the rate of exchange of the copper ion.



#### **CHAPTER 2**

#### LITERATURE REVIEW

# 2.1 THE TRANSITION METAL

# 2.1.1 The first transition metals series

These metals – titanium (Ti), vanadium (V), Chromium (Cr), manganese (Mn), iron (Fe), Cobalt (Co), and nickel (Ni) - are all major industrial importance.

Scandium is soft, silvery-white metal. It is quite common, there being almost as much of the element in the earth crust as there is of arsenic and almost twice as much as there is of Boron. There are few rich mineral sources of scandium. It usually mixed with other land lanthanides in the minerals thortveitite and weikite. Sometimes it is found in tin and tungsten ores. In all, it is found in over 800 minerals, although only in very small amounts. Scandium is not separated easily from other elements. It is separated using a special and difficult process involving an exchange of ions (electrically-charged atoms) of the elements while in solution. Scandium is employed mostly high-intensity lights. Astronomers have also found that some stars contained much scandium (Fetzer, 1992).



Titanium is light weight, silver gray metal. It is widely distributed in the earth crust, being the ninth most abundant element. However it is never found in the pure state, usually occurring in the mineral ilmenite or rutile. Titanium resists corrosion and rust better than stainless steel. It can be drawn in fine wire and is not affected by strong acids in with stand temperature up to about 427°C. It is also has higher strength-weight ratio then steel. However it is expensive to extract titanium from the ores in which it is found. For this reasons, the metals is not widely used, being restricted to aircraft and jet engines. It also used in manufacture of white paints, plastics, paper, and porcelains enamels (Fetzer, 1992).

Vanadium is silver-white metal that is found very small quantities throughout the earth crusts. It is used mostly in manufacturer of ferrovanadium, in an alloy of iron and 50 to 70 percent vanadium. This strong rust-resistance alloy is used in production of steel parts for airplanes, cars and locomotive, and for making high speed cutting tools. It also suitable for use in nuclear reactors. Vanadium is an important trace element in human body (Fetzer, 1992).

Chromium is a glossy, gray, fairly soft metal. It is found in nature usually combined with the oxygen and iron in a mineral called chromites. Sometimes it is called chrome; the metal does not easily corrode and become very shiny when polished. For these reasons, chromium is often used to plate metals used for car bumpers, door handles and decorative trim. Stainless steel contains at least 10 percents chromium. Stainless steels because it doesn't easily rust, is often used to making eating utensils and kitchen equipment. Chromium-steel alloys are used to make ball-bearing,



safes, armor plating for ships and military vehicles, and the cutting edges of high speed machines tools.

Manganese is brittle, silver gray metals, plentiful throughout the earth crust, though not always accessible. The lead producers of the metal are Soviet Union, South Africa, and Brazil. Manganese is employed mainly in special steels is used in making heavy duty machinery, safes, electromagnet (for radar transmitter and computer storage units), and stainless steels. It is used in the manufactured dry cell batteries, dyes, paints, fertilizers and compound for water purifications. Manganese is a trace element required by all plants and animals, including human being (Fetzer, 1992).

Iron, a silvery-white metal in its pure state, is found in nature combined in others elements in iron ores. It is very abundant throughout the earth crust. Iron is by far the most commonly used structural metal. It is often combined with another metal of this series into steel (the most common), alloy steel, stainless steel, and tools steel. Iron is indispensable to human body. The average adult male body contain one-eight ounce (3.5 grams). About 65% is found in hemoglobin, which carries oxygen from the lungs to the various parts of the body. Iron is also needed for the proper functioning of cells, muscles and other tissues (Fetzer, 1992).

Cobalt is hard, silver-white metal that is also magnetic, like iron and nickel. It is relatively rare in the earth crust. In the making of steel alloys, cobalt's ability to withstand high temperature makes it ideal for use in gas turbines, jet engines, and other equipment that operates at high temperatures. Cobalt is also alloyed with aluminum



and nickel or iron for use in magnets in radio, TV sets, and others devices. An isotope of cobalt-cobalt 60-is use in treatment of cancer (Fetzer, 1992).

Nickels are malleable white metal that is magnetic, can be polished to a high gloss, and does not tarnish easily or rust. The leading nickel producers are the Soviet Union, Canada and Australia. Nickels resistance to corrosion makes it invaluable in making storage batteries. Nickel-iron alloys are used to make Armor plate and machines parts. Invar (an alloy of nickel, iron and other metals) is used for meter scales and pendulum rods, because it expands very little with temperature changes. Nickel-silver is alloy used in tableware. The U.S. five cent piece, also called a nickel, is made of copper and nickel (Fetzer, 1992).

Zinc is shiny, bluish –white metal that is hard and brittle in room temperature. Zinc was first smelted in China and India about 1000A.D. The technology for making zinc appeared in west in the 18<sup>th</sup> century when the metal was called Indian tins or calamine. Zinc is comparatively rare in nature, although widely distributed, occurring mainly combination with sulphur in mineral called sphalerite, or zinc blended. It is never found in pure state in nature. It is the 4<sup>th</sup> most common metal in industrial use (after iron, copper and aluminum) zinc rarely used alone, but large amounts are employed in alloys. In die casting (using molds to form objects out of liquid metal), zinc is combined with aluminum and small amounts of copper and magnesium. To make brass, 3 to 45 percent of zinc is combined with copper. The addition of tin to zinc and copper make bronze. In galvanizing, steel is hot dipped in molten zinc. The thin layer of zinc protects the seal from corrosion. Galvanized metal is used in such products as roof gutters and tank linings. Zinc alloys are used to protect ships and



buried steel structures. United States pennies are made mostly from zinc alloys with a thin coating copper (Fetzer, 1992).

Zinc is also used in making paint pigment, cosmetics, soaps, skin ointments, and various plastics. A compound of zinc is used to coat inside of television screens and florescent lamp. Other compounds of zinc are used in dry batteries and in embalming and to protect wood from decay and from insects. Zinc is essentials trace element in the human body in the red blood cells. It is also an important ingredient in of the hormone insulin. In plants and animal, zinc is necessary for normal growth and healing. Its deficiency can cause leaf disease in trees (Fetzer, 1992).

# 2.2 COORDINATION COMPLEXES AND LIGANDS

# 2.2.1 Coordination Complexes

Coordination compounds, such as the FeCl<sup>4<sup>-</sup></sup> ion and CrCl<sub>3.6</sub> NH<sub>3</sub>, are called such because they contain ions or molecules linked, or coordinated, to a transition metal. They are also known as complex ions or coordination complexes because they are Lewis acid-base complexes (Baba, 1994). The ions or molecules that bind to transition metal ions to form these complexes are called ligands (from Latin, "to tie or to bind"). The number of ligands bound to the transition metal ion is called the coordination number (Sharpe, 1981).



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