

SEPARATION OF METAL IONS (COPPER AND CHROMIUM) USING POLY
(HYDROXAMIC ACID) CHELATING RESIN

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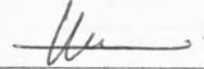
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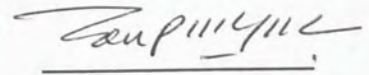
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ABSTRACT

Resins are insoluble polymeric matrix containing mobile ions that are capable of exchanging with ions in the surrounding medium. Two types of polymerization are known, which are step-reaction polymerization and chain-reaction polymerization. Poly(hydroxamic acid) chelating resin was prepared from the reaction of poly(methyl acrylate) grafted sago starch with hydroxylamine. The copolymer was obtained by free-radical initiating process which is a chain-reaction polymerization that uses an initiator, with hydroxylamine as the initiator. Such polymerization is used because the radicals produced are tolerant of many functional groups and solvents. The weight of the resin produced was 27.53 g. The resin was used for the separation of metal ions, which are copper and chromium from mixed metal ion solution that contained two different metal ions, which are Cu-Cr. The metal ion separation was done using the column method. The affinity of the resin towards copper and chromium ions can be determined from the metal ions absorption by the resin. From the experiment, it is known that the poly(hydroxamic acid) chelating resin absorbed more Cu^{2+} ions than Cr^{3+} . As a conclusion the poly(hydroxamic acid) chelating resin have a higher affinity towards Cu^{2+} than Cr^{3+} .



**PEMISAHAN ION LOGAM (KUPRUM, ZINK, KROMIUM)
MENGUNAKAN RESIN POLY(HYDROXAMIC ASID)**

ABSTRAK

Resin adalah matrik polimer tidak larut yang mengandungi ion-ion bebas yang berkemampuan untuk bertukar dengan ion-ion di persekitarannya. Terdapat dua jenis pempolimeran yang diketahui, iaitu pempolimeran *step-reaction* dan pempolimeran *chain-reaction*. Resin poly(hydroxamic acid) telah disediakan daripada tindakbalas antara kanji sagu poly(methyl acrylate) dengan larutan hydroxylamine. Copolimer tersebut diperolehi daripada proses pencetusan radical bebas yang mana merupakan suatu pempolimeran *chain-reaction* yang menggunakan pencetus, di mana pencetus yang digunakan adalah larutan hydroxylamine. Pempolimeran tersebut digunakan kerana radikal-radikal yang dihasilkan mudah menerima kebanyakan kumpulan berfungsi dan pelarut. Jumlah resin yang dihasilkan adalah 27.53 g. Resin tersebut telah digunakan dalam pemisahan ion-ion logam, iaitu kuprum dan kromium daripada larutan campuran ion-ion logam yang mengandungi dua ion logam yang berlainan, iaitu Cu-Cr. Pemisahan ion logam tersebut dilakukan menggunakan kaedah *column*. Tahap pemilihan ion untuk membentuk kompleks bagi resin tersebut boleh ditentukan daripada peyerapan ion-ion logam oleh resin. Daripada eksperimen, telah diketahui bahawa resin poly(hydroxamic acid) meyerap lebih cenderung untuk menyerap ion Cu^{2+} berbanding ion Cr^{3+} . Boleh disimpulkan bahawa resin poly(hydroxamic acid) mempunyai tahap pemilihan ion yang lebih tinggi terhadap ion Cu^{2+} daripada ion Cr^{3+} .



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LIST OF SYMBOLS

C	celcius
cm	centimeter
g	gram
L	liter
mg	miligram
M	molar
μm	micrometer
mL	milliliter
n	moles
ppm	part per million
\rightleftharpoons	Reversible reaction



CHAPTER 1

INTRODUCTION

1.1 Introduction

Ion exchange was originally discovered to take place in soils. The first commercially available ion exchanger was amorphous alumino-silicate gels. The instability of these gels toward acid solutions and their variable behaviour led chemists to seek alternatives. This search eventually resulted in the synthesis of organic ion exchange resins, which soon dominated the field because of their uniformity, chemical stability and control of ion exchange properties through synthetic methods (Abe *et al.*, 1991).

Poly(hydroxamic acid) chelating resin is an ion exchange resin capable of forming complexes with a wide range of metal ions. It contains hydroxamic group that have a high affinity towards individual metal ions, which make it widely used for metal extraction, separation, recovery, microelements extraction and other potential applications. Its high selectivity to certain metal ions made it suitable to be used as the ion exchanger for extracting copper, iron and zinc metal ions from complex solutions. This ability made it widely used in the recovery of metal ions from various mediums; for example, extracting uranium from sea water. It have been prepared by various



methods, such as from copoly(acrylamide divinylbenzene), from an acrylonitrile-divinyl benzene mixture, from poly(methyl acrylate) (Lutfor *et al.*, 2000) and others.

The purpose of this study is to determine the selectivity of the resin towards some metal ions by using the column method. In this study, the resin will be used to separate several pairs of metal ions from a sample solution containing two different metal ions. From the separation, the selectivity of the resin towards the selected metal ions can be determined by calculating the metal ion capacity absorbed by the resin.

The resin will be prepared from poly(methyl acrylate) grafted copolymer and hydroxylamine with the presence of alkaline. The metal ion separation will be carried out using the column method. The analysis of the experiment will be done using the atomic absorption spectrophotometer (AAS).



1.2 Objectives of study

The objectives of this study are:

- a) To prepare the poly(hydroxamic acid) chelating resin.
- b) To determine the metal ions separation ability of the poly(hydroxamic acid) chelating resin.
- c) To calculate the capacity of the metal ions absorbed by the resin.



CHAPTER 2

LITERATURE REVIEW

2.1 Resin

2.1.1 Ion Exchange Resin

Ion exchange resins (resin) are insoluble polymeric matrix containing mobile ions capable of exchanging with ions in the surrounding medium. Each resin has a different number of mobile ion sites that set the maximum quantity of exchanges per unit of resin. The backbone of insoluble polymers is made from crosslinked polystyrene, polyacrylate esters and polymethacrylate esters. These polymers are crosslinked with a poly-functional monomer to give the insolubility effect in solvents, physical and chemical stability. Ion exchange resins are classified into two groups which are cation exchangers, which have positively charged mobile ions available for exchange, and anion exchangers, whose exchangeable ions are negatively charged. Both anion and cation resins are produced from the same basic organic polymers. The difference between them is the ionizable group attached to the hydrocarbon network. This functional group determines the chemical behaviour of the resin. Resins which are cation exchangers can be classified into strong or weak acid cation exchangers while



anion exchanger resins are classified into strong or weak base anion exchangers (Bolto and Pawlowski, 1987).

Strong acid resins are named so because their chemical behavior is similar to strong acids. The resins are highly ionized in both the acid (R-SO₃H) and salt (R-SO₃Na) form. They can convert a metal salt to the corresponding acid by the reaction:



In a weak acid resin the ionizable group is a carboxylic acid (COOH) as opposed to the sulfonic acid group (SO₃H) used in strong acid resins. These resins behave similarly to weak organic acids that are weakly dissociated. Weak acid resins show a much higher affinity for H⁺ than strong acid resins. This characteristic allows for regeneration to the hydrogen form with significantly less acid than is required for strong acid resins. Almost complete regeneration can be accomplished with stoichiometric amounts of acid. The degree of dissociation of a weak acid resin is strongly influenced by the solution pH. Consequently, resin capacity depends in part on solution pH. Figure 2.1 shows that a typical weak acid resin has limited capacity below a pH of 6.0 making it unsuitable for deionizing acidic metal. The ion exchange resin used in most metal recovery systems is a weak acid resin (Schweitzer, 1979).



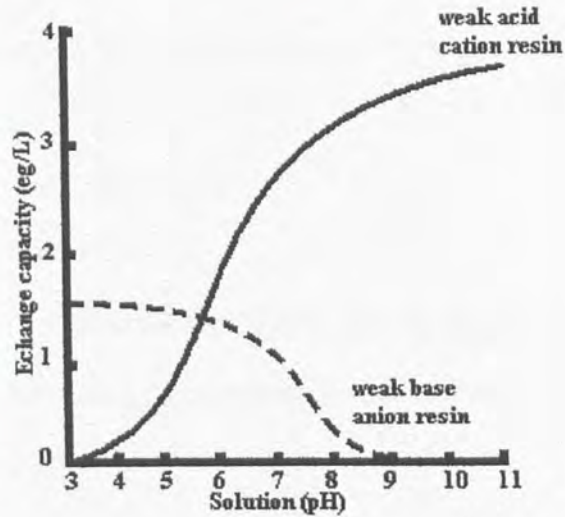


Figure 2.1 Exchange capacity of weak acid and base resin (Schweitzer, 1979).

Like strong acid resins, strong base resins are highly ionized and can be used over the entire pH range. These resins are used in the hydroxide (OH) form for water deionization. They will react with anions in solution and can convert an acid solution to pure water, with the reaction as follows:



Regeneration with concentrated sodium hydroxide (NaOH) converts the exhausted resin to the hydroxide form (Schweitzer, 1979).

Weak base resins are like weak acid resins in that the degree of ionization is strongly influenced by pH. Therefore, weak base resins show minimum exchange capacity above a pH of 7.0 (Figure 2.1). Weak base resins are preferred over strong

base resins because they require less regenerant chemical. A reaction between the resin in the free base form and HCl would proceed as follows:



The weak base resin does not have a OH⁻ form as the strong base resin does. Therefore regeneration needs only to neutralize the absorbed acid, it need not provide OH⁻ (Schweitzer, 1979).

Ion exchange resins are stable over the full range of pH. They are almost stable to most inorganic or organic chemicals except for strong oxidants such as dissolved chlorine, ozone or peroxides. The resin matrix is decrosslinked and becomes soft. The rate of oxidation is enhanced by the increase of the concentration of oxidants and by the presence of metals such as iron and copper which serve as catalysts at high temperatures (Horwitz *et al*, 1993).

Resins that are currently available show a range of selectivity's and thus have broad application. Selectivity of ion exchange resins in order of decreasing preference is shown in Table 2.1.



Table 2.1 Selectivity of ion exchange resins.

Strong acid cation exchanger	Strong base anion exchanger
Barium	Iodide
Lead	Nitrate
Calcium	Bisulfite
Nickel	Chloride
Cadmium	Cyanide
Copper	Bicarbonate
Zinc	Hydroxide
Magnesium	Fluoride
Potassium	Sulfate
Ammonia sodium	
Hydrogen	

2.1.2 Total Exchange Capacity

The total available capacity or total exchange capacity of the resin is a measure of all the functional groups on a resin and is reported on a unit volume of wet resin or a unit weight of dry resin. Test methods are available to measure the total exchange capacity of each type of ion exchange resin. The total exchange capacity is not necessarily the same as the operating capacity. The actual operating capacity is usually lower than the total capacity for an ion exchange process such as in recovery or purification applications. Some of these factors affecting the operating capacity are total and



relative ion concentrations, charge density of ions, flow rate, temperature, pH, regeneration efficiency and the design of the equipment used (Horwitz *et al.*, 1993).

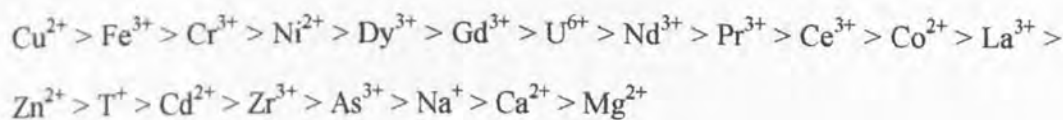
2.2 Poly(hydroxamic acid) chelating Resin

Chelating resins are ligands (electron donor atom, anion) with coordinating atom which when bonded to a metal, a ring compound will be formed. Chelating resins behave similarly to weak acid cation resins but show a high degree of selectivity for heavy metal cations. Chelating resins have special functional groups which contain two or more electron donor atoms that can form coordinate bonds to a single metal atom. Coordinate bonds are formed when shared pair of electrons is contributed by only one of the atoms, which is the donor. Classes of chelating functional groups of industrial importance are phosphonic acids, amino-, carboxylic acids and sulfur compounds. Resins that have metal chelating capabilities include those containing aminophosphonic acid or iminoacetic acid or thiol sites. Chelating resins contain specially oriented ionic and non-ionic groups that selectively capture specific ions from complex mixtures. This makes the resin has a hierarchy of preferences, for example, it prefers Cu^{2+} ions more than Co^{2+} ions. A characteristic feature of the chelating resin is the preservation of their high selectivity with respect to individual ions in the presence of large quantities of other ions. The apparent selectivity for a given metal depends upon concentration, the presence of other species, and pH. The affinity of the resin for a given metal can be increased or decreased by adjusting the pH. To obtain the desired pH, buffer solutions are preferably used (Marcus and Marinsky, 1993).



The high degree of selectivity for heavy metals permits separation of these ionic compounds from solutions containing high background levels of calcium, magnesium, and sodium ions. A chelating resin shows greater selectivity for heavy metals in its sodium form than in its hydrogen form. Regeneration properties are similar to those of a weak acid resin; the chelating resin can be converted to the hydrogen form with slightly greater than stoichiometric doses of acid because of the fortunate tendency of the heavy metal complex to become less stable under low pH conditions. The disadvantage of using heavy-metal-selective chelating resins is the high cost (Marcus and Marinsky, 1993).

Polymeric chelating resins are able to bind heavier transition, lanthanide and actinide metal ions in the presence of calcium, magnesium, sodium and potassium. Poly(hydroxamic acid) chelating resin has potential commercial importance with the knowledge that hydroxamic groups are able to form complexes with wide range of metal ions. This resin exhibits high affinity for copper, iron, chromium, nickel, dysprosium, lanthanum and uranium. It is used for separation of some transition metal ions from their binary mixture (Lutfor *et al.*, 2001). The sorption of metal ions by the resin was pH dependant and its selectivity towards these metal ions is in the following order:



2.2.1 Reported synthesis methods

Poly(hydroxamic acid) resin can be synthesized by various methods and for various purposes. Some of the reported methods are from copoly(acrylamide divinylbenzene) by treatment with hydroxylamine hydrochloride in the presence of potassium hydroxide (Lutfor *et al.*, 2000), by polymerizing an acrylonitrile-divinyl benzene mixture. (Lutfor *et al.*, 2001), from poly(methyl acrylate) and hydroxylamine in the presence of an alkaline medium, and from Amberlite IRC-50 by conversion of the carboxylic acid to acid chloride or to an ester and nitrile (Lutfor *et al.*, 2000) followed by treatment with hydroxylamine.

2.2.2 Advantages

The poly(hydroxamic acid) chelating resin that was used in this study was made from poly(methyl acrylate) grafted sago starch. The preparation using the poly(methyl acrylate) grafted copolymer is said to be efficient, cost effective and simple work. The sago starch is abundant and a renewable source. It is said that the poly(hydroxamic acid) chelating resin is the efficient resin for extracting heavy transition and other metal ions (Lutfor *et al.*, 1999). The resin preserves their high selectivity with respect to individual ions in the presence of large quantities of other ions. This is important to extract microelements from a complex solution (Lutfor *et al.*, 2001).



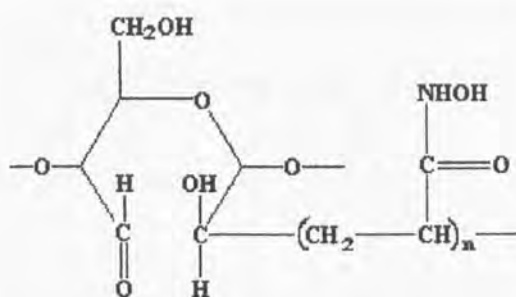


Figure 2.2 Poly(hydroxamic acid) chelating resin.

2.2.3 Applications

The poly(hydroxamic acid) chelating resin can be used in various purpose. Some of its applications are for recovering metals from metal salts and the recovery of uranium from from simulated sea water (Lutfor *et al.*, 2000), for the recovery of silver and gold at trace levels. Gold was recovered from neutral and acid solutions of up to 1.0 M acid concentration. Silver and gold separation was achieved by selective desorption of silver with 0.5 M nitric acid. Gold may only be quantitative removed by the use of potassium cyanide as eluent (Lutfor *et al.*, 1999). For the separation of some transition metal ions from their binary mixture, for binding capacities of copper, iron, chromium, nickel, dysprosium, gadolinium and uranium were found excellent, other metal ions have significant sorption capacities (Lutfor *et al.*, 2001). For extracting a complex of metal ions from various sources. The design of such chelating resin depends on the stability of the basic macromolecular network, which is suited to the desired chelating functional groups for specific metal ions. Polymeric chelating resins are able to bind heavier transition, lanthanide and actinide metal ions in the presence of calcium, magnesium, sodium and potassium. For studying the binding properties of

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