SORPTION STUDIES OF Cu$^{2+}$ ION ON VARIOUS SOLID MATRICES SUCH AS GEL AND MEMBRANES

Honors Project

In fulfillment of the Requirements for

The University Honors College

University of North Carolina at Pembroke

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December 5, 2005

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Date: 12-05-05

Date: 12/15/05

Date: 4/19/06
SORPTION STUDIES OF Cu\(^{2+}\) ION
ON VARIOUS SOLID MATRICES
SUCH AS GEL AND MEMBRANES

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June 1, 2005 – August 5, 2005

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Abstract

In this study it is intended to decontaminate Cu$^{2+}$ ion from drinking water. Simple chromatographic experiments were conducted with various solid matrices such as iminodiacetic acid (IDA) gel and various IDA membranes such as Sartobind and Convective Interactive Media (CIM) disk. Elution conditions and flow rate were optimized for better performance of the separation process. It is clear from these studies that the membranes do have larger binding capacity of Cu$^{2+}$ ion over the gel under similar conditions. Also, membranes are easier to operate than the gel. Finally, affinity constant, KD, between the Cu$^{2+}$ ion and the IDA gel was determined using Langmuir adsorption isotherm. This study proved that the drinking water containing Cu$^{2+}$ ion even at lower concentration such as 10 ppm could be decontaminated by theses solid matrices.

Introduction

Copper is necessary for the biological system to function. Copper is a vital nutrient needed daily to prevent anemia and keep the skeletal, reproductive, and nervous systems healthy. For adults, it is essential to take 1-5 mg of copper daily. The sources of copper are fish, soybeans, coffee, tea, cocoa, liver, drinking water (copper plumbing). Copper deficiency could result in gastrointestinal symptoms such as nausea, abdominal pain, diarrhea, vomiting (3). Copper deficiency has also been linked to more serious situations such as connective tissue being abnormal, central nervous system disorders, but it is rare in humans.

Copper toxicity is result of greater concern. Some of the symptoms associated with excessive copper intake are liver cirrhosis in children and also Alzheimer’s (5,6). Recent studies
have shown that copper in drinking water promotes an Alzheimer's-like pathology in rabbits. When this same study was conducted later at another laboratory the rabbits did not exhibit an Alzheimer's-like pathology. This disparity was eventually traced to the fact that the rabbits were drinking distilled water when previously they had been drinking tap-water (5). If this study proves true for humans as well copper consumption could be a tremendous health threat for numerous people.

Unfortunately, the elevated levels of copper have also been linked to harming shellfish, finfish, and other aquatic organisms (2). Copper has also been known to be linked to more long-term consequences such as DNA damage or mutagenesis (1). Excessive copper intake has also been known to be involved in cancer and can be a risk factor in estrogen-dependent cancers. Research has proven that there is a 72% increase in the copper content of malignant tumors of the ovary, uterus, and cervix. Other studies have also shown high copper contents in breast cancers (4). Copper can also interfere with brain chemicals, the neurotransmitters, which is a cause of depression, anxiety, aggressive behavior and memory loss. Research has shown that learning disabled, hyperactive, retarded and autistic children have elevated copper levels. More research will help solve many of these problems by removing excess copper from drinking water by allowing only an adequate amount of Cu$^{2+}$ to enter the body.

Attention will be paid on developing techniques to separate Cu$^{2+}$ ion from both drinking water and waste water. In this study various solid matrices such as IDA gel, Sartobind IDA membrane, Sartobind epoxy membrane and CIM disk coupled with IDA will be used to decontaminate Cu$^{2+}$ ion from water. Factors such as flow rate and the presence of other ions will also be considered.
Materials

In this experiment, the equipments and materials used BIO-RAD Econo Pump, BIO-RAD model 2110 Fraction Collector, BIO-RAD silicon tubing (1.6mm); microfuge tubes, 10, 20 mL test tubes, 10, 100, 250, volumetric flasks, Gilson P5000, P1000, P200, P100, P20, P10 Pipetman Pipets, Fisherbrand transfer pipet, Kontes Flex Column™, Branson Ultrasonicator, Fisher Scientific Traceable® stopwatch, 10, 50,150, 250, 4000 ml beakers, 50,125, 225 Pyrex Erlenmeyer flask, 10ml syringes with luer lock, rubber tubing, stop cork, Fisher-Brand Redi-Tip™ General Purpose pipet tips, 1000 mL plastic bottles, test tube racks, metal spatulas, Barnstead Thermolyne Cimarec® Stirer, Drummonds Scientific Co. pipet-aid, pipet glass, parafilm, Varian SpectraAA 55B Atomic Absorption Spectrometer, Varian Cu²⁺, Zn²⁺ glass cathode tubes, Acetylene gas and compressed air supplied by MWSC purity gases, filter adapter, Pyrex 30ml frit filter, 6M HCl, 3M HNO₃, Ethanol, Acetone, Mettler Toledo Balance PB 303-S, Fisher Scientific Accumet Research pH meter (AR 10), Fisher Scientific stir bar, Fisher Scientific Cupric Nitrate with a formula weight of 232.59 g, Cu(NO₃)₂ • 2½H₂O, Fisher Scientific Calcium Chloride Anhydrous with a formula weight of 110.99g CaCl₂, Fisher Scientific Hydroxylamine Hydrochloride with a formula weigh of 69.49g, NH₂OH • HCl, Fisher Scientific Magnesium Chloride with a formula weight 203.31g, MgCl₂ • 6H₂O, Fisher Scientific Sodium Hydroxide with a formula weight 40.00g, NaOH, Fisher Scientific Zinc Sulfate with the formula weight of 287.54g, ZnSO₄ • 7H₂O, Sigma® Ethylenediaminetetraacetic Acid (EDTA) with a formula weight of 416.2 g, C₁₀H₁₂N₂O₆Na₂ • 2H₂O, Sigma® Iminodiacetic Acid (IDA) with a formula weight of 133.1g, Sigma® Chelating Resin (Iminodiacetic Acid), Sigma® Sodium Chloride with a formula weight of 58.44g,
Calculation

Making 10 ppm Cu$^{2+}$ solution out of Cu(NO$_3$)$_2$$ \cdot $ 2$H_2$O

\[
(0.010 \text{ g Cu}^{2+} \times 232.59 \text{ g Cu(NO}_3)_2$$ \cdot $ 2$H_2$O) / 63.546 \text{ g Cu}^{2+} = 0.0366 \text{ g/L Cu(NO}_3)_2$$ \cdot $ 2$H_2$O
\]

Making 1 mM of EDTA (Ethylenediamine-tetraacetic Acid)

\[
(0.001 \text{ mol EDTA} \times 416.2 \text{ g EDTA}) / 1 \text{ mol EDTA} = 0.416 \text{ g/L EDTA}
\]

Making 10 mM of EDTA (Ethylenediamine-tetraacetic Acid)

\[
(0.010 \text{ mol EDTA} \times 416.2 \text{ g EDTA} \times 500 \text{ ml}) / (1 \text{ mol EDTA} \times 1000) = 2.081 \text{ g EDTA}
\]

Making 100 mM of EDTA (Ethylenediamine-tetraacetic Acid)

\[
(0.100 \text{ mol EDTA} \times 416.2 \text{ g EDTA} \times 500 \text{ ml}) / (1 \text{ mol EDTA} \times 1000) = 20.81 \text{ g EDTA}
\]

Making 10 mM of CaCl$_2$ (Calcium Chloride)

\[
(0.010 \text{ mol} \times 110.984 \text{ g} \times 1000 \text{ ml}) / 1000 \text{ ml} = 1.110 \text{ g/L CaCl}_2
\]

Making 100 mM (0.1M) NaCl (Sodium Chloride)

\[
(0.100 \text{ mol} \times 58.450 \text{ g}) = 5.845 \text{ g/L NaCl}_2
\]

Making 10 mM of MgCl$_2$$ \cdot $ 6$H_2$O (Magnesium Chloride)

\[
(0.010 \text{ mol} \times 95.211 \text{ g} \times 1000 \text{ ml}) / 1000 \text{ ml} = 0.952 \text{ g/L MgCl}_2
\]

Making 10 mM of ZnSO$_4$$ \cdot $ 7$H_2$O (Zinc Sulfate)

\[
(0.010 \text{ mol} \times 287.54 \text{ g}) / 65.39 \text{ g} = 0.444 \text{ g/L MgCl}_2
\]

Making cleaning solution by using 1 part of HNO$_3$ and 2 part of HCl
30ml of Copper 10ppm
(10mg / 1L) * (1L / 1000ml) = 0.01 mg/ml
30ml * (0.01mg/1ml) = 0.30mg * 1000 = 300ug of Copper injected

\[ x = \frac{y - 0.0014}{0.0992} \]
\[ x \text{ is the concentration} \]
\[ y \text{ is the absorbance} \]

Quantity = (3.75ml * x)
3.75ml is the calibrated volume
\[ x \text{ is the concentration} \]

Copper retained (ug) = add all of the Quantity together / 1000

**Reagents**

- **1mM EDTA solution** was made by dissolving 0.416 g of EDTA into 1000mL of distilled-deionized water.
- **10mM EDTA solution** was made by adding 2.081 g of EDTA into 500mL of distilled-deionized water.
- **10ppm Cu\(^{2+}\) solution** was made by adding 0.036 g of Cupric Nitrate into 1000mL of distilled-deionized water.
- **Cu\(^{2+}\) solution** was made by adding 0.036 g of Cupric Nitrate, 5.845g of Sodium Chloride into 1000mL of Tap water.
- **10ppm Zinc solution** was made by adding 0.044g of Zinc Sulfate into 1000mL of distilled-deionized water.
- **Zinc solution** was made by adding 0.044g of Zinc Sulfate, 0.036g of Cupric Nitrate, 5.845g of NaCl into 1000mL of distilled-deionized water.
- *Zinc solution* was made by adding 0.044g of Zinc Sulfate, 0.036g of Cupric Nitrate into 1000mL of distilled-deionized water.

- *10mM Calcium Chloride* was made by adding 1.110 g of Calcium Chloride into 1000mL of distilled-deionized water.

- *Calcium Chloride solution* was made by adding 1.110g of Calcium Chloride, 0.036g of Cupric Nitrate, 5.845g of NaCl into 1000mL of distilled-deionized water.

- *Calcium Chloride solution* was made by adding 1.110g of Calcium Chloride, 0.036g of Cupric Nitrate into 1000mL of distilled-deionized water.

- *10mM Magnesium Chloride* was made by adding 0.952g of Magnesium Chloride into 1000mL of distilled-deionized water.

- *Magnesium Chloride solution* was made by adding 0.952g of Magnesium Chloride, 0.036g of Cupric Nitrate, 5.845g of NaCl into 1000mL of distilled-deionized water.

- *Magnesium Chloride solution* was made by adding 0.952g of Magnesium Chloride, 0.036g of Cupric Nitrate into 1000mL of distilled-deionized water.

- *Stock A* was made by adding 174.2g of Potassium Phosphate (K₂HPO₄) in 1 Liter of DDW.

- *Stock B* was made by adding 136.09g of Potassium Phosphate Primary Standard (KH₂PO₄) in 1 Liter of DDW.

- *Cleaning solution* was made by 20ml 3M HNO₃ and 20ml 6M HCl, dilute this solution with distilled-deionized water (wear gloves to protect hands).

- *Adsorption Isotherm*
  - Using 11 test tubes label from 0 to 1.
  - The total volume of the test tubes equals 10 ml.
  - The test tube number indicates the concentration of copper in ppm.
  - Remove 3 ml from each tube for the control or initial concentration.
  - Then remove the remaining 7ml and place it into 10 ml beakers.
  - Add 1ml of chelating resin with iminodiacetic acid (IDA) into 10 ml beaker with 7ml of copper solution.
  - Afterwards place the 10ml beakers onto a stirring plate for 15 minutes.
- When the stirring is complete the supernatant is removed leaving only the chelating resin with IDA gel in the beaker.
- All the gel is then placed into one 50 ml beaker together.
- Then 40ml of 100mM Ethylenediamine-Tetraacetic acid (EDTA) is put into the beaker with the chelating resin with IDA gel.
- The beaker is place on a stirring plate and allowed to stir for 30 minutes at a speed of 6.
- After stirring is completed we removed the EDTA from the chelating resin with IDA gel.
- The chelating resin with IDA gel is put in a Pyrex 30 ml Frit Filter which is placed on top of a 500 ml Pyrex Filter Flask which is attached to a vacuum.
- The Frit Filter must be previously thoroughly cleaned with acetone and ethanol.
- The chelating resin with IDA gel is then washed with 3000ml of distilled-deionized water.
- Lastly the clean gel is placed back into the refrigerator until next use.

- **Making Standard Copper Solution**

  - Clean six test tubes:
  - Label B, 2, 4, 6, 8, 10.
  - Put 10ml of DDW into all test tubes except test tube label 10
  - Remove 2ml DDW for test tube 2, and add 2ml of 10ppm Copper solution.
  - Remove 4ml DDW for test tube 4, and add 4ml of 10ppm Copper solution.
  - Remove 6ml DDW for test tube 6, and add 6ml of 10ppm Copper solution.
  - Remove 8ml DDW for test tube 8, and add 8ml of 10ppm Copper solution.
  - For test tube 10, add 10ml of 10ppm Copper solution.

- Other standard solutions are prepared the same way as the Standard Copper Solution but instead of using copper solution just use the other solution.
Methods

Sartobind® epoxy-activated membrane

The Sartobind® Epoxy 75 was immobilized with Iminodiacetic Acid (IDA) as the ligand by coupling the 50ml stock A and adding about 2ml of stock B until it reach pH of 8 for about 3 hours. The membrane area is 75 cm² or 2.1 ml volume. The Sartobind® Epoxy 75 was connected to BIO-RAD Econo Pump by BIO-RAD silicon tubing. Prior to all of the experiment, distilled-deionized water (DDW) was used to flush all of the air out the system and any left over IDA to ensure of the consistency of the fraction volumes. 30mL of Cu²⁺ solution was passed through the module collected by the fraction collector. Then 60mL of DDW was passed through to collect any unbound or loosely bound cation metal. 50mL of EDTA solution was passed through the module to elute any chelated metal from the membranes, and collected by the fraction collector. This process is called chelation. 60mL of DDW was passed through to wash the membrane and this is being collected by the fraction collector, some more wash are done but are discard. All of these solutions were passed through the module at the rate of 1mL per minute. Fraction was collected at the rate of 5mL/ min, but the calibrated value was at 3.75mL / min for the first fraction collector. The second fraction collector was calibrated at the rate of 5mL / min.

Sartobind® IDA 75

Sartobind® IDA 75 already has the Iminodiacetic acid (IDA) as the ligand on the membrane. The membrane area is 75 cm² or 2.1 ml volume. The flow rate improve with the higher ionic strength, therefore all of the solution are being run with Sodium Chloride (NaCl). Distilled-
deionized water (DDW) is being replace by the Sodium Chloride. The Sartobind® IDA 75 was connected to BIO-RAD Econo Pump by BIO-RAD silicon tubing. Prior to all of the experiment, Sodium Chloride was used to better the flow rate and to equilibrate the membrane to ensure of the consistency of the fraction volumes. 30mL of Cu$^{2+}$ solution was passed through the module collected by the fraction collector. Then 60mL of NaCl was passed through to collect any unbound or loosely bound cation metal. 50mL of EDTA and NaCl solution was passed through the module to elute any chelated metal from the membranes, and collected by the fraction collector. This process is called chelation. 60ml of NaCl was passed through to wash the membrane and this is being collected by the fraction collector, some more wash are done but are discard. All of these solutions were passed through the module at the rate of 1mL per minute. Fraction was collected at the rate of 5mL/ min, but the calibrated value was at 3.75mL / min for the first fraction collector. The second fraction collector was calibrated at the rate of 5mL / min.

**Chelating Resin with Iminodiacetic Acid**

Chelating Resin with Iminodiacetic Acid was pack into the Kontes Flex Column™. 30mL of Cu$^{2+}$ solution was passed through the module collected by the fraction collector. Then 60mL of DDW was passed through to collect any unbound or loosely bound cation metal. 50mL of EDTA solution was passed through the module to elute any chelated metal from the membranes, and collected by the fraction collector. This process is called chelation. 60ml of DDW was passed through to wash the membrane and this is being collected by the fraction collector, some more wash are done but are discard. All of these solutions were passed through the module at the rate of 1mL per minute. Fraction was collected at the rate of 5mL/ min, but the calibrated value was at
3.75mL / min for the first fraction collector. The second fraction collector was calibrated at the rate of 5mL / min. Adsorption Isotherm is also being done using Chelating Resin with IDA.

**Convective Interaction Media Iminodiacetic Acid Disks (CIM® IDA DISKS)**

CIM® IDA Disk is immobilize with IDA as the chelating ligand. The disk is 12mm in diameter, 3mm thick, and the bed volume is 0.34ml. 30mL of Cu²⁺ solution was passed through the module collected by the fraction collector. Then 60mL of DDW was passed through to collect any unbound or loosely bound cation metal. 50mL of EDTA solution was passed through the module to elute any chelated metal from the membranes, and collected by the fraction collector. This process is called chelation⁴. 60ml of DDW was passed through to wash the membrane and this is being collected by the fraction collector, some more wash are done but are discard. All of these solutions were passed through the module at the rate of 1mL per minute. Fraction was collected at the rate of 5mL / min, but the calibrated value was at 3.75mL / min for the first fraction collector. The second fraction collector was calibrated at the rate of 5mL / min.

Sartobind® epoxy-activated membrane, Sartobind® IDA 75, Chelating Resin with Iminodiacetic Acid, CIM® IDA disks are run with Magnesium Copper, Tap Water Copper, Zinc Copper, and Calcium Copper.

All of these fractions were analyzed by Atomic Absorption Spectrometer. These fractions were used to compare to the standard solution prepared prior to the analysis.
Results and Discussion

Effect of Cu\textsuperscript{2+} adsorption on various solid matrices

Figure 1, shows how copper interact with various matrices such as Epoxy with IDA membrane, Sartobind with IDA membrane, and CIM Disk with IDA membrane. This figure showed CIM Disk with IDA has higher affinity for Cu\textsuperscript{2+} ion than the other two membranes.
Adsorption capacity of different matrices on various sorbents

Figure 2 really showed how CIM Disk with IDA have the greatest capacity to bind with metal ion, while IDA membrane is still a better adsorption material than the other two membrane.
Various concentration of EDTA on Chelex 100

Figure 3 is showing 10mM EDTA is less material being use compared to 100mM EDTA, in term of cost 10mM EDTA is cheaper and it worked as effective as the 100mM EDTA.
Effects of flow rate on Chelex 100

Figure 4 proved with the flow rate of 1.0 ml/min will be as effective as the flow rate of 0.5 ml/min. This means work can get done fast and effective.
Effect of various ions with Cu$^{2+}$ adsorption on Chelex 100

Figure 5a show with the influence of Ca$^{2+}$, Mg$^{2+}$, Tap water on Cu$^{2+}$ ion upon the gel, Chelating Resin with IDA, is not very much. With these metal ion act with Cu$^{2+}$ ion the affect is not very noticeable. This proved that this gel can be use under various conditions.
Effect of various ions with Cu\textsuperscript{2+} adsorption on Sartobind IDA-75

Figure 5b shows the influence of Ca\textsuperscript{2+}, Mg\textsuperscript{2+}, Tap water on Cu\textsuperscript{2+} ion upon the membrane, Sartobind IDA - 75, is not very much. With these metal ion act with Cu\textsuperscript{2+} ion the affect is not very noticeable. This proved that this membrane can be use under various conditions.
Effect of various ions with Cu$^{2+}$ adsorption on CIM DISK with IDA

Figure 5c show with the influence of Ca$^{2+}$, Mg$^{2+}$, Tap water on Cu$^{2+}$ ion upon the membrane, CIM Disk with IDA, is not very much. With these metal ion act with Cu$^{2+}$ ion the affect is not very noticeable. This proved that this membrane can be use under various conditions.
**Cu^{2+} preferential adsorption on various matrices**

Figure 6 shows the various solid matrices preferred binding to Cu^{2+} ion versus other metal ion in this case Zn^{2+} ion. This figure also once again shows CIM Disk with IDA have the highest binding capacity with metal ion than other solid matrices.

![Graph showing preferential adsorption of Cu^{2+} and Zn^{2+}](image)

**Figure 6**

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Determination of affinity constant by Langmuir Adsorption Isotherm

Adsorption Isotherm of Cu²⁺ on Chelating Resin (Chelex 100)

Figure 7a

Double reciprocal plot

\[ y = 0.0051x + 1.3941 \]

\[ R^2 = 0.9854 \]

Figure 7b
Since the objective of this work is to decontaminate Cu²⁺ ions both from drinking and waste water, sorbents such as gel and membranes were tested as solid matrices. As mentioned earlier, these sorbents were activated by appropriate chemistry and coupled with iminodiacetic acid (IDA) which is the ligand that coordinates with Cu²⁺ ion. As described in the experimental section the chromatogram was performed on all the sorbents such as Chelex 100 resin, Sortobind IDA membrane, Sartobind epoxy membrane and CIM Disk. The results are as shown in Fig.1. It is clear from these results that all the membranes could decontaminate Cu²⁺ ion from water even if the concentration is very low in the order of 10 ppm. At the end of each cycle of adsorption and desorption the solid matrices could be regenerated for the next cycle of operation.

In order to get an idea of the efficiency of the solid matrices a comparison was performed among the gel and the membranes with respect to the binding capacity of Cu²⁺ ion per ml of the gel and per ml of the membrane under similar conditions. The results are as shown in Fig.2. From this result it is very obvious that the binding capacity of CIM disk is 5 times more than the Sartobind IDA membranes and 30 times more than the Chelex 100 resins. This result obviously indicates that the membrane has a superior surface area compared to that of the gel. In other words for better separation the contact area between the solution and the solid matrix should be large. The advantage of the membranes over the gel is the membranes are significantly easy to handle. On the other hand the gel has to be repacked perfectly occasionally for proper and continuous performance.
It was essential in this study to optimize operating conditions such as the elution condition of Cu2+ ions from water and the flow rate. Therefore experiments were carried out to optimize those conditions and the results are as shown in Fig.3 and Fig. 4. It is evident from these results that the most effective elution condition is 10 mM EDTA. Regarding the flow rate the gel behaves almost the same way both at 0.5 and 1.0 ml/min.

In order to see the influence of other cations such as Ca2+, Mg2+, and tap water, experiments were conducted in the presence of these ions under similar conditions. The results from Fig.5a, Fig.5b and Fig.5c indicate that these ions and the tap water do not have any influence at all. Notably membranes behaved much better than the gel. Among the membranes, results show that the CIM disk performance is much better than the Sortobind membranes.

Just out of curiosity, experiments were carried out to see the preferential adsorption of Cu2+/Zn2+ ion over these sorbents under the given condition of equal concentration mixtures of both these ions in water. The results are as indicated in Fig. 6. From these results it is obvious that all the sorbents prefer Cu2+ ion over Zn2+ ion. Also it is clear that the CIM disk prefers Cu2+ ion 2 times more than the Zn2+ ion and is exactly the same with the other sorbents. This is because the coordination chemistry of Cu2+ ion is different than the coordination chemistry of Zn2+ ion.

Finally to understand about the affinity of Cu2+ ion over IDA, experiments were carried out several times to determine the affinity constant, KD, between the IDA gel and the Cu2+ ion.

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Therefore as described in the experimental section an adsorption isotherm was performed on this gel with various concentration of Cu2+ ion with a given amount of the gel. The results are as shown in Fig. 7a and 7b. From Fig. 7a, it is very clear that the adsorption isotherm follows Langmuir pattern which is

\[ Q_a = \frac{QX C}{(K_D + C)} \]

Where \( Q_a \) is the quantity of Cu2+ ion bound per ml of the gel at equilibrium
\( QX \) is the maximum amount of Cu2+ ion could bind per ml of the gel
\( C \) is the equilibrium concentration of Cu2+ ion
\( K_D \) is the dissociation constant (affinity constant between the IDA gel and the Cu2+ ion)

A linear plot was drawn between \( 1/Q_a \) and \( 1/C \) to determine \( K_D \). The ratio of the slope/y-intercept will give the \( K_D \) value and it is 0.0036 ppm.

**Conclusions**

1. It is easy to operate membranes over the gel.
2. Membranes do have larger surface areas therefore the binding capacity of Cu2+ ion is better than the IDA gel under similar conditions.
3. With membranes work can be performed at higher flow rates.
4. Elution condition is moderate with 10 mM EDTA.
5. In comparison CIM disk performance is much better than the other membranes and membrane performance is much better than the gel.
6. These membranes work well under any condition, meaning at the real condition of decontamination of water containing Cu2+ ions.

7. Membranes and gel prefer Cu2+ ion over the Zn2+ ions under the equimolar concentration mixture of Cu2+ / Zn2+ ions.
References


Acknowledgements

I wish to thank Dr. Siva Mandjiny for his guidance and support in the completion of this project. I like to thank Ms. Fallon Lowery for allowing me to share her data. I would also like to thank Dr. Paul Flowers, whom hard work and dedication to get this program started at the University of North Carolina at Pembroke and ensure the survival of the program on this campus, Mrs. Shanna Harrelson, and Ms. Carolyn Parsons for their assistance and instruction with laboratory instruments and supplies. This summer research experience is supported by the University of North Carolina at Pembroke and is funded by the National Science Foundation Research Experiment for Undergraduates.

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