

**REMOVAL OF HAZARDOUS OXYANION POLLUTANTS BY
LANTHANUM (III) HYDROXIDE AND LANTHANUM (III) -LOADED
ION EXCHANGERS**

By

YEHYA M. AHMED

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia in Fulfilment of the Requirements
for the Degree of Master of Science**

December 2004

DEDICATION

To:

My father's soul, my dear mother and brothers for their pray for me. My beloved wife for her unlimited sincere sacrificing, encouragement and support during my study. My beloved brave sons, I will never forget your braveness.

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of requirements for the degree of Master of Science

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LANTHANUM (III) HYDROXIDE AND LANTHANUM (III) – LOADED
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Chairman: Associate Professor Md Jelas Haron, Ph.D.

Faculty : Science

La(III) loaded poly(hydroxamic acid) chelating resin (LaPHA), lanthanum hydroxide-loaded poly(hydroxamic acid) chelating resin (LaOHPHA) and lanthanum hydroxide ($\text{La}(\text{OH})_3$) were prepared and evaluated as sorbents for removal of As(V), As(III) and Cr(VI) from aqueous solution. LaPHA was prepared by loading lanthanum(III) onto PHA resin which was prepared by the reaction of PMA with hydroxyl amine hydrochloride in alkaline medium. LaOHPHA was prepared by treating LaPHA with ammonia solution and $\text{La}(\text{OH})_3$ was prepared by neutralizing LaCl_3 solution with ammonia solution.

In order to understand the sorption characteristics of As(V), As(III) and Cr(VI) by the sorbents, batch and column studies were performed under various experimental conditions. The parameters studied were pH, contact time, isotherm study,

temperature, sorbent dosage, presence of competitive anions, flow rate, sorption-desorption study and industrial wastewater treatment.

The results of batch study indicated that the sorption process was pH dependent and endothermic. Kinetic study showed that uptake of As(V) ion by all of the sorbents reached equilibrium after about 15 hours and about 10 hours for Cr(VI) by La(OH)₃. The maximum sorption capacity of LaPHA for As(V) at room temperature determined from the Langmuir isotherm was 14.8 mg-As/g-sorbent compared to that of LaOHPHA for As(V) which was 17.7 mg-As/g-sorbent, while the maximum sorption capacity of La(OH)₃ for As(V) was 133.3 mg-As/g-sorbent and for Cr(VI) was 33.1 mg-Cr/g-sorbent. The results also showed that the La(OH)₃ in the PHA resin (LaOHPHA) was more stable in acidic medium compared to the La(OH)₃. The uptake increased with increasing sorbent dosage and also increased by increasing in temperature. In the presence of competitive ions, metal uptake decreased and it was found that phosphate ion is the most effective one.

In desorption study, As(V) could be removed almost quantitatively from LaPHA and LaOHPHA by eluting with 0.4M and 0.2M NaOH solutions, respectively. The column with LaPHA could be used for not more than 3 cycles and 2 cycles for LaOHPHA. Both LaPHA and LaOHPHA resins were found effective to remove arsenic from wastewater samples from wood treatment industry. The percentages of arsenic removal from wastewater by the sorbents were higher for lower

concentrations samples. More than 90% removal was obtained from the sample with the lowest arsenic concentration.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
bagi memenuhi keperluan untuk ijazah Master Sains

**PENYINGKIRAN POLUTANT OKSIANION BERBAHAYA OLEH
LANTHANUM (III) HIDROKSIDA DAN LANTHANUM (III)
DIPEGUNKAN DALAM PENUKAR ION**

Oleh

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Resin pengkelat poli(asid hidrosamik) dipegunkan La(III) (LaPHA), poli(asid hidrosamik) dipegunkan La(OH)₃ (LaOHPHA), dan La(OH)₃ telah disediakan dan dikaji keberkesanannya untuk penyingkiran ion logam As(V), As(III) dan Cr(VI) dari larutan akueus. Resin PHA telah disediakan melalui tindakbalas PMA dengan hidrosil amin hidroklorida di dalam medium berkali. LaPHA disediakan dengan tindakbalas lanthanum (III) dengan larutan ammonia.

Untuk memahami ciri-ciri erapan LaPHA, LaOHPHA dan La(OH)₃ terhadap As(V), As(III) dan Cr(VI), kajian kelompok dan turus telah dijalankan di bawah pelbagai keadaan eksperiment. Parameter yang dikaji pH, masa, isoterma, dos pengrap,

kehadiran ion-ion saingan, kadar aliran, erapan-penyaherapan dan rawatan terhadap sample air buangan industri.

Keputusan kajian kelompok menunjukkan bahawa proses erapan dipengaruhi oleh pH dan bersifat endotermik. Kajian kinetic menunjukkan keseimbangan tindakbalas bagi erapan As(V) dan Cr(VI) masing-masing oleh diperolehi selepas 15 jam dan 10 jam. Kapasiti erapan maksimum untuk LaPHA pada suhu bilik yang ditentkan daripada isoterma Langmuir adalah 14.8 mg-As/g-pengerap berbanding oleh LaOHPHA sebanyak 17.1 mg-As/g-pengerap manakala bagi La(OH)₃ sebanyak 133.3 mg-As/g-pengerap dan bagi Cr(VI) sebanyak 33.1 mg-As/g-pengerap. Erapan meningkat dengan peningkatan dos pengerap. Kehadiran ion-ion saingan akan mengurangkan erapan logam dan ion fosfat adalah yang paling effective.

Dalam kajian penyaherapan, hamper semua As(V) dapat disingkirkan secara kuantitatif dari LaPHA dan LaOHPHA dengan larutan pengelusi masing-masing iaitu larutan 0.4 M dan 0.2 M NaOH. Turus LaPHA dapat digunakan tidak lebih daripada 3 kali manakala turus LaOHPHA dapat digunakan tidak 2 kali sebelum kecekapan turus menurum kepada kira-kira 50%. Kedua-dua resin LaPHA dan LaOHPHA telah didapati berkesan untuk merawat air buangan industri dari industri perawatan kayu. Peratus penyingkiran arsenic adalah bergantung kepada kepekatan asal ion arsenic dalam sample. Lebih daripada 90% arsenic telah berjaya disingkirkan dari sample air buangan dengan kepekatan arsenic paling rendah.

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I certify that an Examination Committee met on 10 December 2004 to conduct the final examination of Yehya M. Ahmed on his Master of Science thesis entitled “Removal of Hazardous Oxyanion Pollutants by Lanthanum (III) Hydroxide and Lanthanum (III) Loaded Ion Exchanger” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

YEHYA M. AHMED

Date:

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LIST OF ABBREVIATIONS

PHA	Poly (hydroxamic acid)
PMA	Poly methyl acrylate
$t_{1/2}$	Time to remove 50% of the initial concentration of the metal ion solution
K_s	Rate constant
LH	Lanthanum hydroxide
LC	Lanthanum carbonate
BLC	Basic lanthanum carbonate
U. S. EPA	United States Environmental Protection Agency
LaPHA	Lanthanum (III) loaded-poly (hydroxamic acid) resin complex
LaOHPHA	Lanthanum hydroxide loaded- poly (hydroxamic acid) resin
FTIR	Fourier transforms infrared spectroscopy
XRD	X-Ray Diffraction
CHNS	Carbon, hydrogen, nitrogen and sulphur
DVB	Divinyl benzene
d	The basal spacing
n	An integer (1, 2, 3...)
λ	Wave length \AA°
θ	The angle of incidence of the x-ray beam and the atomic planes
q_e	The amount of the metal sorbed at equilibrium (mg/g).
q	The amount of the metal sorbed at time (mg/g).
t	Time of equilibrium(min)
C_e	Equilibrium concentration of the metal ion (mg/L)
Q_m	Maximum sorption capacity of the system (mg/g)
b	Constant related to the energy of the sorption (L/mg)
K_d	Distribution coefficient defined as N_e/C_e
T	Absolute temperature (K)
R	Gas constant
ΔS°	Entropy change (J/mol K)
ΔH°	Enthalpy change (J/mol)
ΔG°	Free energy change (J/mol)
K_{sp}	Solubility constant

CHAPTER I

INTRODUCTION AND LITERATURE REVIEW

1.1 ION EXCHANGERS

Ion exchangers are solid and suitably insolubilized high molecular weight polyelectrolyte which can exchange their mobile ions for ions of equal charge from the surrounding medium. The resulting ion exchange is reversible and stoichiometric with the displacement of one ionic species by another on the exchanger. Viewed in different light, ion exchangers can be considered high molecular weight acids or bases with a high molecular weight cation, which can exchange their hydrogen or hydroxyl ions for equally charged ions and thus are converted into high molecular weight salts.

1.1.1 Synthetic Resin Ion Exchangers

Synthetic ion exchange resins are insoluble poly electrolytes with limited moisture regain capacity. On the bases of their function, there are three types of ion exchangers, cation exchanger, anion exchanger and amphoteric ion exchanger (Dorfner, 1973). Polymerization and polycondensation in principle can be used as the synthesis routes to form the resin matrix. At the present time, polymer resins have become more important than polycondensation resins. They have a higher chemical

and thermal stability. The most important starting material, which is used nearly exclusively in exchangers of the strongly acid and strongly basic types produced on a large scale, is styrene. The styrene is cross-linked with itself and with divinylbenzene into a polymeric network (Figure 1.1) (Dorfner, 1973).

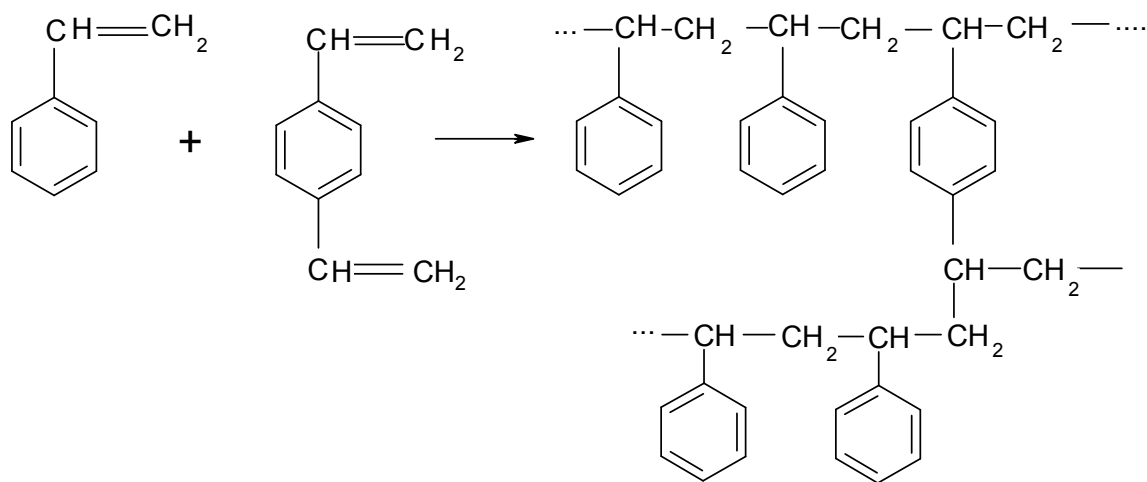


Figure 1.1: Cross-linking of styrene with divinylbenzene into a polymeric network (Dorfner, 1973).

On the other hand, matrices for weakly acid cation exchangers are formed, which is also crosslinked with divinylbenzene, from acrylic acid or methacrylic acid (Figure 1.2) (Dorfner, 1973).

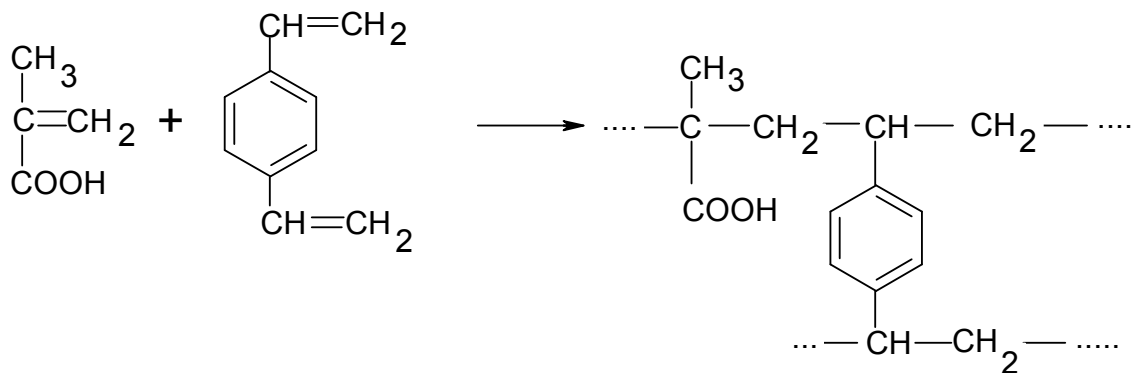


Figure 1.2: Formation of weakly acid cation exchanger from the reaction between methacrylic acid and divinylbenzene (Dorfner, 1973).

In polycondensation resins, the matrix is usually constructed from phenol and formaldehyde (Figure 1.3) (Dorfner 1973):

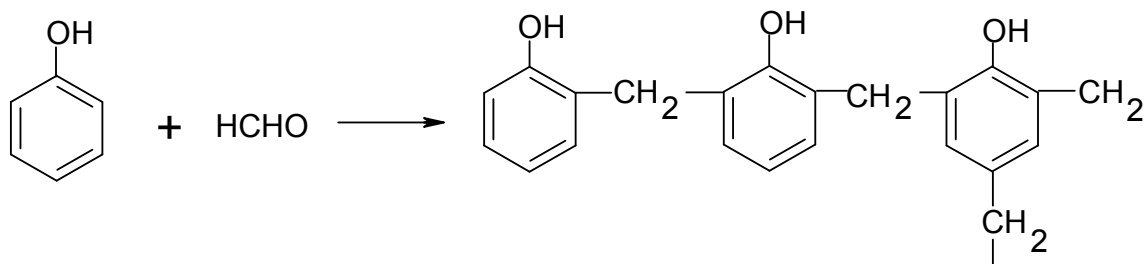


Figure 1.3: Formation of poly condensation resins from phenol and formaldehyde (Dorfner, 1973).

The properties of the exchanger depend on the nature of the phenols, the quantity of starting materials used, and their side groups (Vasilev, 1968).

1.1.2 Specific and Chelating Ion Exchangers

Specific ion exchangers are those types in which functional groups were introduced which have the properties of a specific reagent. The specificity is based on the chemical structure of the ion exchanger itself and may not be confused with the phenomenon of selectivity. Because of its specificity, an ion exchanger can sorb one ionic species to the exclusion of others under a broad range of conditions.

Specific ion exchangers include the entire group of chelating resins whose specificity is based on the chelate or complex-forming functional group. The best known commercial type is the iminodiacetate ion exchanger (Dowex A – 1), in which the iminodiacetate groups are directly attached to the styrene matrix (Figure 1.4):

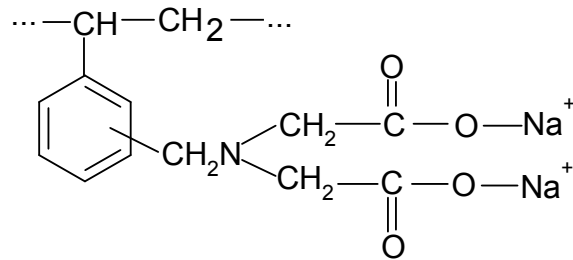


Figure 1.4: The structure of iminodiacetate chelating ion exchanger (Dowex A – 1) (Dorfner, 1973).