SYNTHESIS AND CHARACTERISATION OF METAL ION-IMPRINTED POLYMERS FOR SEPARATION OF SELECTIVE RARE EARTH ELEMENTS

NIK ROHANI BINTI NIK MUSTAPA

Master of Science

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

We hereby declare that we have checked the thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

____

(Supervisor's Signature)

Full Name : PROF. DR. MASHITAH MOHD YUSOFF

Position : PROFESSOR

Date :

____

(Co-supervisor's Signature)

Full Name : PROF. DR. MD. LUTFOR RAHMAN

Position : PROFESSOR

Date :

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citation which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang.

____

(Author's Signature)

Full Name : NIK ROHANI BINTI NIK MUSTAPA

ID Number : MKT 13002

Date :

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Faculty of Industrial Sciences & Technology

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FEBRUARY 2018

DEDICATION

This thesis dedicated to my beloved husband, family and friends,
for their endless support and encouragement
throughout my journey to complete this research.

ACKNOWLEDGEMENTS

First of all I would like to thank Allah S.W.T., the Almighty for giving me the strength, passion and patience to finish my research and the thesis.

Special thanks and deepest appreciation to my supervisors, Professor Dato' Dr. Mashitah binti Mohd. Yusoff and Professor Dr. Md. Lutfor Rahman for providing such a wonderful opportunity to pursue this endeavor—their help and guidance were invaluable. With their leadership, patience and generosity enabled me to complete the works during my master program. I feel extremely fortunate to have spent my master research with them. Thanks also to senior lecturer, Mr. Muhammad Norfazli Abd. Malek, for the best support, advice and knowledge during my studies.

I would like to express my sincere thanks to faculty's technical staff, Mr. Shaharunizam Umar, Mr. Muhamad Husaini Sulaiman, Mr. Mohd Farid Jaafar, Mr. Khairul Affendi Yusof, Mr. Muhammad Halim Paboh, Mrs. Nordiana Hazani and Mrs. Norshahida Zaidon who assist with various instruments for data collection. Also thanks to all Central Laboratory's technical staff.

Especially my sincere indebtedness, my deepest gratitude goes to my beloved parents for their deeply love and endless support, prayers and encouragement throughout my life. I am also grateful to my husband, Mr. Mohd Izzat Fida'iy Mohamed and also my kids for their love, patience, and understanding that were inevitable to make this research success with flying colours.

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LIST OF SYMBOLS

C_i	Concentration of metal ions before extraction
C_f	Concentration of metal ions after extraction
C_t	Metal concentration at time t
K	Selectivity coefficient
K_d	Distribution ratio
$K_d^{Ln^{3+}}$	Distribution ratios of lanthanides
$K_d^{M^{n+}}$	Distribution ratios of foreign metal ions
M	Mass of L-IIPs materials
q_t	Adsorption amount at time t
Q	Sorption capacity
V	Volume of the solution

LIST OF ABBREVIATIONS

BAL	Bronchoalveolar lavage
CP	Control polymer
FAAS	Flame atomic absorption spectrometry
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
GFAAS	Graphite furnace atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ICP-MS	Inductively coupled plasma-mass spectrometry
IIP	Ion-imprinted polymers
ISE	Ion-selective electrode
LLE	Liquid-liquid extraction
MIIP	Metal ion-imprinted polymers
MIPs	Molecular imprinted polymers
NAA	Neutron activation analysis
REE	Rare earth elements
SPE	Solid phase extraction
USA	United States of America
UV-vis NIR	Ultraviolet-visible Near-Infrared

LIST OF CHEMICAL COMPOUNDS

AIBN	2,2-azobisisobutyronitrile
Al	Aluminium
Ca	Calcium
Ce	Cerium
CeO_2	Cerium(IV) oxides
$CHCl_3$	Chloroform
CMA	bis(carboxymethyl)amino
D_2EHPA	bis(2-ethylhexyl)phosphoric acid
DCQ	5,7-dichloroquinoline-8-ol
DVB	Divinylbenzene
EGDMA	Ethylene glycol dimethacrylate
EtOH	Ethanol
Eu	Europium
Fe	Iron
Gd	Gadolinium
H_2SO_4	Sulphuric acid
HCl	Hydrochloric acid
La	Lanthanum
Lu	Lutetium
MeOH	Methanol
MnO_4^-	Permanganate ion
Mo	Molybdenum
Nb	Niobium
Nd	Neodymium
PAA	Polyacryloylacetone
PC88A	2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester
Pm	Promethium
Pr	Praseodymium
Sm	Samarium
Tb	Terbium
Ti	Titanium
THF	Tetrahydrofuran
TMPTM	Trimethylolpropane

Yb	Ytterbium
Zn	Zinc
Zr	Zirconium

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ABSTRAK

Polimer tercetak ion (IIPs) telah menerima banyak perhatian dalam pelbagai bidang kerana keupayaannya yang tinggi dalam memilih sasaran ion logam. Dalam kajian ini, polimer tercetak ion-lanthanida (L-IIPs) telah disintesis dengan melarutkan sejumlah stoikiometri ion lantanum dan ejen pengkompleks terpilih ligan Schiff atau azobenzene dan juga sebatian indol dengan pelarut bersama ejen ditautsilang (ethylene glycol dimethacrylate (EGDMA)), monomer berfungsi (4 vinylpyridine), dan bahan pemula (2,2-azobisisobutyronitrile (AIBN). Rongga dalam bahan polimer yang sepadan dengan ion lanthanida khusus terhasil dengan melarutkan polimer menggunakan larutan HCl akueus selepas pempolimeran. Penyingkiran lengkap ion lanthanida dari L-IIPs telah dibuktikan dengan ICP-MS, FESEM dan analisis keadaan pepejal oleh spektroskopi UV-Vis NIR. FT-IR membuktikan proses penghasilan sebatian kompleks di antara L-IIPs dan ion lanthanida berlaku melalui nitrogen, oksigen hidroksil dan sulfur dari ligan Schiff, asas ligan azobenzene dan sebatian indol. Pada optima (pH 6), kapasiti penyerapan maksimum yang dicapai oleh L-IIPs adalah La-IIP –Schiff (25.0 mg g^{-1}), La-IIP-Azo (24.3 mg g^{-1}), Ce-IIP-Schiff (24.5 mg g^{-1}), Ce-IIP-Azo (24.7 mg g^{-1}), Pr-IIP-Schiff (125.3 mg g^{-1}), Nd-IIP-Schiff (126.5 mg g^{-1}), Sm-IIP-Schiff (127.6 mg g^{-1}), Eu-IIP-Schiff (128.2 mg g^{-1}) dan Gd-IIP-Schiff (129.1 mg g^{-1}). Dalam kajian selektif, L-IIPs menunjukkan selektif yang baik kepada ion nadir bumi praseodymium, gadolinium, europium, samarium, cerium, neodymium dan lanthanum ion dalam kehadiran kation, promethium. Keputusan menunjukkan bahawa bahan tercetak lebih cenderung untuk menarik beberapa ion lanthanida berbanding ion pesaing logam lain dengan cas yang sama dan radius ionik yang hampir serupa. L-IIPs yang disintesis telah diguna semula untuk sebanyak 8 kali tanpa penurunan ketara afiniti pengikatan polimer.

ABSTRACT

Ion imprinted polymers (IIPs) have received much attention in diverse fields owing to their high selectivity for targeted metal ions. In the present study, lanthanide-ion imprinted polymers (L-IIPs) were synthesised by dissolving stoichiometric amounts of lanthanum ion and selected complexing agents either Schiff bases, azobenzene base ligands and also indole compounds with solvent in the presence of cross-linking agent (ethylene glycol dimethacrylate (EGDMA)), functional monomer (4-vinylpyridine), and initiator (2,2-azobisisobutyronitrile (AIBN)). The cavities in the polymer materials corresponding to the specific lanthanide ions were created by leaching the polymer using aqueous HCl solution after polymerization. The complete removal of the lanthanide ions from the L-IIPs were confirmed by ICP-MS, FESEM and solid state analysis by UV-vis NIR spectroscopy. FTIR study confirmed the complexation between the L-IIPs and lanthanide ions through the nitrogen, hydroxyl oxygen and sulphur of Schiff base, azobenzene base ligands and indole compounds. At optimum (pH 6), the maximum sorption capacity achieved by the L-IIPs are estimated to be La-IIP-Schiff (25.0 mg g^{-1}), La-IIP-Azo (24.3 mg g^{-1}), Ce-IIP-Schiff (24.5 mg g^{-1}), Ce-IIP-Azo (24.7 mg g^{-1}), Pr-IIP-Schiff (125.3 mg g^{-1}), Nd-IIP-Schiff (126.5 mg g^{-1}), Sm-IIP-Schiff (127.6 mg g^{-1}), Eu-IIP-Schiff (128.2 mg g^{-1}) and Gd-IIP-Schiff (129.1 mg g^{-1}). In the selectivity study, the L-IIPs were found to show good selectivity to the praseodymium, gadolinium, europium, samarium, cerium, neodymium and lanthanum ion in the presence of coexisting cation, promethium. It was found that imprinting results increased the affinity of the material toward several lanthanide ions over other competitor metal ions with the same charge and similar ionic radii. The synthesized L-IIPs were repeatedly used and regenerated for 8 times without a significant decrease in polymer binding affinity.

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