

Synthesis and Characterization of Molecularly Imprinted Polymer for the Removal of Triazine Herbicides from Water Samples

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Synthesis and Characterization of Molecularly Imprinted Polymer for the Removal of Triazine Herbicides from Water Samples

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DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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ABSTRACT

Cyanazine (CYZ), Ametryn (AME) and Atrazine (ATZ) are mostly utilized 1, 3, 5-triazine herbicides to control the growth of broadleaf grasses and weeds, however, their extensive usage has adverse effects on the environment, non-target organisms, and human health. Therefore, molecularly imprinted polymers (MIPs) were applied to remove triazine herbicides in the environment. In this study, MIPs of triazine herbicides were synthesized by precipitation polymerization in the presence of template molecules (CYZ, AME and ATZ), functional monomers (methacrylic acid, acrylamide, 2-vinylpyirdine, and 4vinylpyridine), cross-linker (ethylene glycol dimethacrylate), initiator (azo-bisisobutyronitrile), and solvent (toluene and acetonitrile). For comparison study, the nonimprinted polymers (NIPs) were synthesized without the addition of template molecule. The Fourier Transform Infra-Red (FTIR) analysis of MIPs of triazine herbicides indicated the presence of CYZ, AME and ATZ were observed at ~756 cm⁻¹, ~1450 cm⁻¹, and ~1389 cm⁻¹ ¹, respectively. MIP (AAm) of CYZ had its maximum adsorption efficiency in 6 ppm CYZ solution at pH 7, 0.1 g of polymer and 240 mins of contact time. MIP (MAA) of AME had its highest adsorption efficiency in 7 ppm AME solution at pH 7, 0.1 g of polymer and 210 mins of contact time. MIP (2VP) of ATZ exhibited its maximum adsorption efficiency in 8 ppm of ATZ solution at pH 7, 0.3 g of polymer and 15 mins of contact time. The thermogravimetric analysis (TGA) of MIPs of triazine herbicides depicted that the decomposition of polymers was between ~320 °C to ~500 °C. MIP (AAm) of CYZ, MIP (MAA) of AME and MIP (2VP) of ATZ were more selective towards their target compounds with relative selectivity coefficients of 2.36, 2.66 and 2.77, respectively. The synthesized MIP (AAm) of CYZ, MIP (MAA) of AME and MIP (2VP) of ATZ were successfully applied to remove CYZ, AME and ATZ from different water samples (distilled water, tap water and river water) with removal efficiencies between ~84% to ~95%.

Keywords: Cyanazine, Ametryn, Atrazine, precipitation polymerization, molecularly imprinted polymers

Sintesis dan Pencirian Molekul Polimer Tercetak untuk Penyingkiran Herbisida Triazin daripada Sampel Air

ABSTRAK

Sianazin (CYZ), Ametrin (AME), dan Atrazin (ATZ) adalah herbisida triazin yang banyak digunakan untuk mengawal pertumbuhan rumput dan rumpai daun lebar. Namun, penggunaannya yang berleluasa memberikan kesan buruk kepada alam sekitar, organisma bukan sasaran, dan kesihatan manusia. Oleh itu, molekul polimer tercetak (MIPs) digunakan untuk menyingkirkan herbisida triazin daripada alam sekitar. Dalam kajian ini, MIPs herbisida triazin disintesis menggunakan pempolimeran pemendakan dengan menggunakan molekul templat (CYZ, AME dan ATZ), monomer berfungsi (metakrilik, akrilamida, 2-vinilpiridin dan 4-vinilpiridin), penggabung silang (etilena glikol dimetakrilat), bahan pemula (azo-bis-isobutironitril) serta pelarut (toluen dan asetonitril). Sebagai perbandingan, molekul polimer tidak tercetak (NIPs) disintesis tanpa penambahan molekul templat. Analisa Spektrum Inframerah Transformasi Fourier (FTIR) menunjukkan bahawa kewujudan CYZ, AME dan ATZ dapat dikenalpasti di ~756 cm⁻¹, ~1450 cm⁻¹, dan ~1389 cm⁻¹. Kadar optimum penjerapan maksimum bagi MIP (AAm) CYZ diperolehi dalam larutan CYZ 6 ppm pada pH 7, 0.1 g polimer dengan jangka waktu penjerapan selama 240 mins. MIP (MAA) AME mempunyai kadar penjerapan tertinggi dicapai dalam larutan AME 7 ppm pada pH 7, 0.1 g polimer dengan jangka waktu penjerapan selama 210 mins. MIP (2VP) ATZ mencapai kadar penjerapan maksimum dalam larutan ATZ 8 ppm pada pH 7, 0.3 g polimer dengan jangka waktu penjerapan selama 15 mins. Analisa termogravimetrik (TGA) bagi MIP herbisida triazin menunjukkan penguraian polimer antara ~320 °C to ~500 °C. MIP (AAm) CYZ, MIP (MAA) AME, dan MIP (2VP) ATZ lebih selektif terhadap molekul sasaran dengan koefisien relatif selektif iaitu 2.36, 2.66, dan 2.77, masing-masingnya. MIP (AAm) CYZ, MIP (MAA) AME dan MIP (2VP) ATZ telah berjaya menyingkirkan CYZ, AME dan ATZ dalam pelbagai sampel air (air suling, air paip dan air sungai) dengan kadar penyingkiran antara ~84% to ~95%.

Kata kunci: Cyanazin, ametrin, atrazin, pempolimeran mendakan, molekul polimer tercetak

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LIST OF ABBREVIATIONS

%	Percentage
°C	Degree Celsius
µg/mL	Microgram Per Millilitre
2VP	2-Vinylpyridine
4VP	4-Vinylpyridine
AA	Acrylic Acid
AAm	Acrylamide
ABDV	Azobisdimethylvaleronitrile
Ace	Acetone
ACN	Acetonitrile
AcOH	Acetic Acid
AIBN	Azobisisobutyronitrile
AME	Ametryn
BET	Brunauer-Emmett-Teller
BIPB	Bis-(1-Tert-Butylperoxy)-1-Methylethyl)-Benzene
BPO	Bensoylperoxide
С	Another Kinetic Constant
Ce	Analyte Concentration at Equilibrium
CEC	Capillary Electro Chromatography
C_{f}	Final Concentration in the Solution After Binding
Ci	Initial Concentration in the Solution Before Binding
cm	Centimetre
cm ⁻¹	Per Centimetre

CYZ	Cyanazine
DAD	Diode Array Detection
DCP	Dicumyl peroxide
DEA	Dielectric Thermal Analysis
DIPB	1,3-Diisopropenylbenzene
DIW	Distilled Water
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetry
DSPE	Dispersive Solid-Phase Extraction
DTA	Differential Thermal Analysis
DVB	Divinylbenzene
EDCs	Endocrine Disrupting Chemicals
EDX	Energy Dispersive X-Ray
EGDMA	Ethylene Glycol Dimethacrylate
EPA	Environmental Protection Agency
EtOH	Ethanol
FESEM	Field Emission Scanning Electron Microscopy
FRP	Free Radical Polymerization
FTIR	Fourier Transform Infra-Red
g	Gram
GC	Gas Chromatography
GC-MS	Gas Chromatograph-Mass Spectrometer
GPC	Gel Permeation Chromatography
h	Hour
HEMA	2-Hydroethylmethacrylate

HPLC-UV	High Performance Liquid Chromatography-UltraViolet
IR	Infra-Red
ISEC	Inverse Size Exclusion Chromatography
Κ	Kelvin
k'	Relative Selectivity Coefficient
K1	Pseudo-First-Order
K ₂	Pseudo-Second-Order
K _D	Distribution Ratio
K _{diff}	Pseudo-First-Order Equilibrium Rate Constant
K _F	Maximum Adsorption Capacity of MIP
KL	Langmuir Constant
KPS	Potassium Persulfate
Ksel	Selectivity Coefficient
m	Mass
MAA	Methacrylic Acid
MBAm	N, N-Methylenebisacrylamide
МеОН	Methanol
mg	Milligram
min	Minute
MIP	Molecularly Imprinted Polymer
MISPE	Molecular Imprinted Solid Phase Extraction
MIT	Molecular Imprinting Technology
mL	Millilitre
mmol	Millimoles
mol	Moles

MSPE	Magnetic Solid-Phase Extraction
n	Constant for the Intensity of Adsorption
NIP	Non-Imprinted Polymer
nm	Nanometre
NMR	Nuclear Magnetic Resonance
O/W	Oil-in-Water
PDMS	Diphenyldiethoxysilane
PhTMS	Phenyltrimethoxysilane
ppm	Parts Per Million
qe	Amount of Analyte Adsorbed at Equilibrium Time
q _{max}	Maximum Adsorption Capacity of MIP
q _t	Amount of Analyte Adsorbed at Any Given Time
R _L	Parameter of Equilibrium
RP-HPLC	Reversed Phase-High Performance Liquid Chromatography
rpm	Round Per Minute
SBSE	Stir Bar Sorptive Extraction
SEM	Scanning Electron Microscopy
SERS	Surface-Enhanced Raman Scattering
SPE	Solid Phase Extraction
SPME	Solid-Phase Microextraction
t	Time
TAIC	Triallyl Isocyanurate
TEM	Transmission Electron Microscopy
TEOS	Tetrathoxysilane
TGA	Thermogravimetric Analysis