



UNIVERSITI PUTRA MALAYSIA

***MOLECULARLY IMPRINTED POLYMER FOR THE REMOVAL OF
MERCURY FROM PETROLEUM OIL***

NOR AIN SHAHERA BINTI KHAIRI

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MERCURY FROM PETROLEUM OIL**

By

NOR AIN SHAHERA BINTI KHAIRI

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

August 2016

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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August 2016

Chairman : Nor Azah Yusof, PhD
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Mercury (Hg) is a contaminant in many natural products including hydrocarbon fuels. The presence of mercury can cause problems with downstream processing units as well as safety of workers and environmental issues. Such concern provides incentive to remove the mercury from petroleum oil. Therefore, improvise sorbents is still essential to be further investigated. In recent years, molecularly imprinted polymers (MIPs) have attracted the attention of several researches due to their capability for molecular recognition, easiness of preparation, stability and cost-effective production. By taking advantage of these facts, Hg(II) imprinted polymer and non-imprinted polymer (NIP) were synthesized by bulk polymerization method where cysteine complex (or L-cysteine alone) was polymerized with mercury nitrate (or without it) as a template. Then, methacrylic acid (MAA), 2-hydroxyethyl methacrylate (HEMA), ethylene glycol dimethacrylate (EGDMA) and methanol were added as monomer, co-monomer, cross-linker and solvent respectively. The obtained MIPs were crushed and sieved. The total weight of yield of MIP increased with increasing ratio of monomer, cysteine complex and cross-linker. The temperature used during polymerization was standardized to 70.0°C. The best ratio of monomer to cross-linker is 1:5 while the amount of cysteine complex is 2.0 mmol.

FTIR results displayed the presence of S-H in the cysteine complex but there is no peak of S-H observed in MIP-CC spectrum which conclude the interaction between S-H and Hg(II) has occurred. FESEM shows that MIP possess spherical and densely packed particles with rough surface compared with NIP for both cysteine complex (CC) and L-cysteine(LC). Through BET result, the surface area and pore volume of MIP-CC is larger than NIP-CC while for MIP-LC and NIP-LC is vice versa. From TGA result, monomer MAA fully decomposed at lower temperature (197.0°C) compared with MIP-CC, NIP-CC, MIP-LC and NIP-LC.

The optimum sorption capacity during pH studies was achieved at pH 7 for both MIP-CC and MIP-LC. Besides, the sorption of Hg(II) increased by increasing the dosage of MIP. Both MIP-CC and MIP-LC followed Freundlich isotherm with $R^2=0.9551$ and $R^2=0.9505$ respectively. Meanwhile, the sorption of Hg(II) by MIP is fast within a few second and it is well fitted with pseudo second order reaction. The selectivity result shows that MIP-CC exhibit high selectivity and affinity towards Hg(II) in the presence of competitor ions; Pb(II), Zn(II) and Cd(II) compared with MIP-LC. The reusability of the MIP-CC particles was tested for four times and no significant loss in sorption capacity was observed. From the real samples analysis, the sorption capacities obtained were 93.8% for sludge sample and 29.8% for crude petroleum oil sample with initial Hg(II) concentration of 74.6 $\mu\text{g/L}$ and 1.78 $\mu\text{g/L}$ respectively

A new type of Hg(II) imprinted polymer as a sorbents for removal of mercury from petroleum oil was successfully synthesized. Prepared MIP-CC showed several characteristic such as high thermal stability, fast sorption kinetic and proper selectivity of Hg(II). Thus, MIP-CC can be potentially used as a sorbents for the removal of mercury petroleum oil.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

POLIMER CETAKAN MOLEKUL UNTUK PENYINGKIRAN MERKURI DARIPADA MINYAK PETROLEUM

Oleh

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Merkuri (Hg) adalah bahan cemar di dalam banyak produk semulajadi termasuk minyak hidrokarbon. Kehadiran merkuri boleh menyebabkan banyak masalah dengan unit pemprosesan hiliran serta keselamatan pekerja dan isu-isu alam sekitar. Kebimbangan sedemikian menyediakan insentif untuk menyingkirkan merkuri daripada minyak petroleum. Oleh itu, menambah baik penjerap masih penting untuk kajian lanjut. Dalam beberapa tahun kebelakangan ini, polimer cetakan molekul (MIP) telah menarik perhatian oleh beberapa pengkaji disebabkan oleh keupayaan ia untuk mengenalpasti molekul, kemudahan semasa penyediaan, kestabilan dan kos pengeluaran yang efektif. Dengan mengambil kesempatan daripada fakta-fakta ini, cetakan polimer Hg(II) dan polimer bukan cetakan molekul (NIP) telah disintesis melalui kaedah pempolimeran pukal dimana kompleks sistin (atau L-sistin sahaja) dipolimerkan bersama merkuri nitrat (atau tanpanya) sebagai acuan. Selepas itu, asid metakrilik (MAA), 2-hidrosietil metakrilat (HEMA), etilena gliko dimetilenakrilat (EGDMA) dan metanol ditambahkan sebagai monomer, monomer bersama, penggabung dan pelarut bagi setiap masing-masing. MIP yang diperolehi dengan itu dihancurkan dan ditapis. Jumlah berat hasil MIP bertambah dengan peningkatan nisbah monomer, sistin-kompleks dan penggabung. Suhu yang digunakan semasa polimeran diselaraskan pada suhu 70.0°C. Nisbah yang terbaik bagi monomer dan penggabung ialah 1:5 manakala jumlah sistin kompleks ialah 2.0 mmol.

Hasil FTIR yang diperolehi menunjukkan kehadiran S-H dalam spektrum sistin kompleks tetapi tidak ada puncak S-H diperhatikan dalam spektrum MIP-CC. Kesimpulannya, interaksi antara S-H dan Hg(II) telah berlaku. FESEM menunjukkan bahawa MIP mempunyai zarah sfera dan padat dengan permukaan kasar berbanding dengan NIP bagi kedua-dua sistin kompleks (CC) dan L-sistin (LC). Melalui hasil BET, jumlah luas permukaan dan isipadu liang MIP-CC lebih besar berbanding NIP-CC sementara bagi MIP-LC dan NIP-LC adalah sebaliknya. Daripada hasil TGA, monomer MAA terurai sepenuhnya pada suhu yang rendah (197.0°C) berbanding dengan MIP-CC, NIP-CC, MIP-LC dan NIP-LC.

Kapasiti penjerapan optimum semasa ujian pH telah dicapai pada pH 7 bagi kedua-dua MIP-CC dan MIP-LC. Selain itu, penjerapan Hg(II) meningkat dengan peningkatan dos MIP. Kedua-dua MIP-CC dan MIP-LC mengikut isoterma Freundlich dengan $R^2 = 0.9551$ dan $R^2 = 0.9505$ bagi setiap masing-masing. Sementara itu, penjerapan Hg(II) oleh MIP adalah pantas dalam masa beberapa saat dan ianya sesuai dengan tindak balas tertib kedua. Hasil selektiviti menunjukkan bahawa MIP-CC mempunyai selektiviti yang tinggi dan tertarik kepada Hg(II) dengan kehadiran ion pesaing ; Pb(II), Zn(II) dan Cd(II) berbanding dengan MIP-LC. Kebolegunaan semula zarah MIP-CC telah diuji sebanyak empat kali dan tiada penurunan ketara pada kapasiti penjerapan yang diperhatikan. Daripada analisis sampel yang sebenar, kapasiti penjerapan diperolehi ialah 93.8% bagi sampel mendapan dan 29.8% bagi sampel minyak petroleum mentah dengan bacaan awal kepekatan Hg(II) adalah 74.6 $\mu\text{g/L}$ dan 1.78 $\mu\text{g/L}$ masing-masing.

Hg(II) cetakan polimer yang baharu sebagai penjerap untuk penyingkiran merkuri daripada minyak petroleum telah berjaya disintesis. MIP-CC yang disediakan menunjukkan ciri-ciri seperti kestabilan haba yang tinggi, kinetic penjerapan pantas dan selektiviti terhadap Hg(II). Oleh itu, MIP-CC berpotensi digunakan sebagai penjerap untuk penyingkiran minyak daripada minyak petroleum.

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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	iii
ACKNOWLEDGEMENTS	v
APPROVAL	vi
DECLARATION	viii
LIST OF TABLES	xiii
LIST OF FIGURES	xv
LIST OF ABBREVIATION/NOTATION	xvii
 CHAPTER	
 1 INTRODUCTION	
1.1 Background of Research	1
1.2 Statement of Problem	2
1.3 Objectives	3
 2 LITERATURE REVIEW	
2.1 Mercury	4
2.1.1 Mercury in hydrocarbon	4
2.1.2 Effect of Mercury on Hydrocarbon Stream	5
2.2 General Principle of Molecular Imprinted Polymer	5
2.3 Types of Molecular Imprinting	6
2.3.1 Covalent Imprinting	6
2.3.2 Non-Covalent Imprinting	7
2.4 Basic elements for synthesis of Molecular Imprinting	7
2.4.1 Template	8
2.4.2 Ligand	8
2.4.3 Functional Monomer	9
2.4.4 Cross-linker	9
2.4.5 Solvents	9
2.4.6 Initiator	10
2.5 Types of Polymerization of MIPs	10
2.5.1 Bulk Polymerization Method	12
2.5.2 Precipitation Polymerization Method	12
2.5.3 Suspension Polymerization Method	12
2.5.4 Multi-step Swelling Polymerization Method	13
2.5.5 In-Situ Polymerization Method	13
2.6 MIP sorbents for Solid Phase Extraction (SPE)	13
2.7 Application of Molecular Imprinted Polymers for the Removal of Hg(II)	14
2.8 Removal of Hg(II) by Related Adsorbents	16
2.9 Removal of Hg(II) From Hydrocarbon Stream by Related Sorbents	18

3	MATERIALS AND METHODOLOGY	
3.1	Chemicals	20
3.2	Instruments	21
3.2.1	pH meter	21
3.2.2	Rotary Evaporator	21
3.2.3	Fourier-Transform Infrared Spectroscopy (FT-IR)	21
3.2.4	Field Emission Scanning Electron Microscopy (FESEM)	22
3.2.5	Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)	22
3.2.6	BET Surface Area	23
3.2.7	Thermal Gravimetric Analysis (TGA)	23
3.2.8	Mercury Analyzer	23
3.3	Synthesis of MIP	24
3.3.1	Synthesis of Cysteine-complex (CC)	24
3.3.2	Synthesis of Cysteine-complex-Hg (II)-MIP (MIP-CC)	24
3.3.3	Synthesis of L-Cysteine-Hg (II)-MIP (MIP-LC)	24
3.4	Extraction of the template	25
3.5	Sorption Studies of MIP and NIP	25
3.5.1	Solid Phase Extraction (SPE) Procedure	25
3.5.2	Effect of pH on sorption of Hg (II) by MIP and NIP	25
3.5.3	Effect of Dosage on sorption of Hg (II) by MIP and NIP	26
3.5.4	Isotherm Studies	26
3.5.5	Kinetic Studies	26
3.5.6	Selectivity Studies	26
3.5.7	Reusability Studies	26
3.5.8	Sorption of Hg (II) from Petroleum Oil Sample	27
4	RESULTS AND DISCUSSION	
4.1	Synthesis of Molecular Imprinted Polymers (MIPs) and Non-Imprinted Polymers (NIPs)	28
4.1.1	Optimization of Monomer and Cross-linker Ratio (M:X)	28
4.1.2	Optimization of Cysteine Complex (CC)	30
4.2	Characterization of MIPs and NIPs	32
4.2.1	Fourier Transform Infrared Spectroscopy (FTIR)	32
4.2.1.1	FT-IR Spectra for MIP-CC and NIP-CC	32
4.2.1.2	FT-IR Spectra for MIP-LC and NIP-LC	33
4.2.2	Thermal Stability Studies of MIPs and NIPs	36
4.2.2.1	Thermal Stability for MIP-CC and NIP-CC	38
4.2.2.2	Thermal Stability for MIP-LC and NIP-LC	39
4.2.3	Morphological Study of MIPs and NIPs	41
4.2.3.1	Morphological Study of MIP-CC and NIP-CC	41
4.2.3.2	Morphological Study of MIP-LC and NIP-LC	41
4.2.4	Surface Area and Porosity Analysis of MIPs and NIPs	44
4.2.4.1	Surface Area and Porosity Analysis of MIP-CC and NIP-CC	44

4.2.4.2	Surface Area and Porosity Analysis of MIP-LC and NIP-LC	46
4.3	Adsorption Studies	47
4.3.1	Effect of pH on sorption of Hg(II) for MIP-CC and MIP-LC	47
4.3.2	Effect of Dosage on MIP-CC and MIP-LC	49
4.3.3	Isotherm Studies of MIP-CC and MIP-LC	50
4.3.4	Kinetic Studies of MIP-CC and MIP-LC	54
4.3.5	Selectivity Studies	57
4.3.6	Reusability Studies of MIP-CC	60
4.4	Sorption of Hg (II) from Petroleum Oil by MIP-CC	61
5	CONCLUSIONS	
5.1	Conclusion	63
5.2	Recommendations	64
	REFERENCES	65
	APPENDICES	73
	BIODATA OF STUDENT	84
	LIST OF PUBLICATIONS	85

LIST OF TABLES

Table	Page
2.1 Advantages and disadvantages of different types of Polymerization for MIP Preparation	11
2.2 Mercury Removal System for Hydrocarbon	18
3.1 List of Chemicals	20
3.2 List of Instruments	21
4.1 List of synthesized of MIP-CC and NIP-CC with different ratios of monomer and cross-linker	29
4.2 List of synthesized MIP-CC and NIP-CC by using different amount of cysteine-complex	31
4.3 Average size particles data, surface area and pore volume of the MIP-CC and NIP-CC	43
4.4 Average size particles data, surface area and pore volume of the MIP-LC and NIP-LC	46
4.5 Langmuir and Freundlich isotherm constant for the MIP-CC and MIP-LC	54
4.6 The first-order and second-order kinetic constants for the MIP-CC and MIP-LC	57
4.7 K_d , K and K' values of Zn(II), Cd(II) and Pb(II) with respect to Hg(II) for MIP-CC and MIP-LC	60
A1.1 Optimization of monomer : cross-linker of MIP-CC	73
A1.2 Optimization of monomer : cross-linker of NIP-CC	73
A2.1 Optimization of cysteine complex for MIP-CC	74
A2.2 Optimization of cysteine complex for NIP-CC	74
A3.1 Effect of pH on sorption capacity of MIP-CC	75
A3.2 Effect of pH on sorption capacity of NIP-CC	75
A3.3 Effect of pH on sorption capacity of MIP-LC	76
A3.4 Effect of pH on sorption capacity of NIP-LC	76
A4.1 Effect of dosage of MIP on percentage removal of Hg(II) by MIP-CC	77
A4.2 Effect of dosage of MIP on percentage removal of Hg(II) by MIP-LC	77
A5.1 Effect of concentration on sorption capacity of Hg(II) By MIP-CC	77
A5.2 Effect of concentration on sorption capacity of Hg(II) by MIP-LC	78
A6.1 Effect of time on sorption capacity of Hg(II) by MIP-CC	78
A6.2 Data kinetic study and determination of reaction order of MIP-CC	79
A6.3 Effect of time on sorption capacity of Hg(II) by MIP-LC	79
A6.4 Data kinetic study and determination of reaction order of MIP-LC	80
A7.1 Selectivity study of MIP-CC	80
A7.2 Selectivity study of MIP-LC	81
A8.1 Reusability study of MIP-CC	81

A9.1	Data removal of Hg(II) ions from sludge sample by using MIP-CC	82
A9.2	Data removal of Hg(II) ions from crude oil sample by using MIP-CC	82



LIST OF FIGURES

Figure	Page
2.1 Schematic representation of non-covalent imprinting	6
2.2 Schematic diagram of off-line MISPE	14
4.1 Optimization of monomer (MAA) to cross-linker (EGDMA)	29
4.2 Proposed chemical reaction of preparation of cysteine-complex	31
4.3 Optimization of cysteine-complex	32
4.4 IR spectra for MIP-CC, NIP-CC, and Cysteine-complex (CC)	33
4.5 Proposed scheme for the preparation of MIP-CC with Hg(II)	35
4.6 IR spectra for MIP-LC, NIP-LC and L-cysteine (LC)	36
4.7 Proposed scheme for the preparation of MIP-LC with Hg(II)	37
4.8 TGA plot of MIP-CC, NIP-CC and monomer P(M)	38
4.9 DTG curve for MIP-CC, NIP-CC and monomer P(M)	39
4.10 TGA plot of MIP-LC, NIP-LC and monomer P(M)	40
4.11 DTG curve for MIP-LC, NIP-LC and monomer P(M)	40
4.12 FE-SEM image of MIP-CC and NIP-CC	42
4.13 FE-SEM image of MIP-LC and NIP-LC	43
4.14 Nitrogen adsorption-desorption isotherm of MIP-CC	45
4.15 Nitrogen adsorption-desorption isotherm of NIP-CC	45
4.16 Nitrogen adsorption-desorption isotherm of MIP-LC	46
4.17 Nitrogen adsorption-desorption isotherm of NIP-LC	47
4.18 Effect of pH towards the sorption of Hg(II) for the MIP-CC and NIP-CC	48
4.19 Effect of pH towards the sorption of Hg(II) for the MIP-LC and NIP-LC	49
4.20 Effect of dosage on the percentage removal of Hg(II) by MIP-CC	50
4.21 Effect of dosage on the percentage removal of Hg(II) by MIP-LC	50
4.22 Effect of equilibrium Hg(II) concentration on the adsorption of Hg(II) ions on MIP-CC	51
4.23 Effect of equilibrium Hg(II) concentration on the adsorption of Hg(II) ions on MIP-LC	52
4.24 Langmuir (a) and Freundlich (b) plots for the sorption of Hg(II) by MIP-CC.	53
4.25 Langmuir (a) and Freundlich (b) plots for the sorption of Hg(II) by MIP-LC.	53
4.26 Time dependent adsorption of Hg(II) ions on the MIP-CC	55
4.27 Time dependent adsorption of Hg(II) ions on the MIP-LC	55

4.28	Kinetic model (a) pseudo first order kinetic model and (b) pseudo second order kinetic model for MIP-CC	57
4.29	Kinetic model (a) pseudo first order kinetic model and (b) pseudo second order kinetic model for MIP-LC.	57
4.30	Selectivity Studies of Hg(II) by MIP-CC and NIP-CC in the presence of competitive ions, Zn(II), Cd(II) and Pb(II).	59
4.31	Selectivity Studies of Hg(II) by MIP-LC and NIP-LC in the presence of competitive ions, Zn(II), Cd(II) and Pb(II).	60
4.32	Reusability of the MIP-CC	61
4.33	Sorption capacities for A) Sludge sample and B) crude oil sample	62
A1	Picture of MIP-CC (left) and NIP-CC (right)	83
A2	Picture of real sample; sludge sample (left) and crude oil (right)	83

LIST OF ABBREVIATIONS

ATR	Attenuated Total Reflectance
BET	Brunauer Emmett Teller
CC	Cysteine-complex
DTG	Differential Thermogravimetric Analysis
EGDMA	Ethylene Glycol Dimethacrylate
FTIR	Fourier Transform Infrared
FESEM	Field Emission Scanning Electron Microscopy
HEMA	2-Hydroxylethyl Methacrylate
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
LC	L-cysteine
MAA	Methacrylic Acid
MATAC	2-(Methacryloyloxy)ethyl] Trimethyl Ammonium Chloride
MIP	Molecularly Imprinted Polymer
NIP	Non-molecularly Imprinted Polymer
MIP-CC	Molecularly Imprinted Polymer- Cysteine-complex
NIP-CC	Non-molecularly Imprinted Polymer Cysteine-Complex
MIP-LC	Molecularly Imprinted Polymer-L-cysteine
NIP-LC	Non-molecularly Imprinted Polymer-L-Cysteine
SPE	Solid Phase Extraction
TGA	Thermal Gravimetric Analysis

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Mercury is one of the hazardous heavy metals both to human and ecosystem because it is highly toxic to central nervous system and tends to bio-accumulate in the human body. This will lead to a variety of adverse health effects including neurological, renal, respiratory, immune, dermatologic and reproductive and developmental neurotoxicity (Poulin, Prüss-Üstün, & Gibb, 2008). Mercury exists in various forms which are elemental mercury, inorganic mercury and organic mercury. All these forms have different toxicities and implication for health. According to World Health Organization, WHO guideline values, the limitation of mercury inside water is $1\mu\text{g/L}$ for total mercury, $1\mu\text{g/m}^3$ mercury in air. In addition, WHO estimated a tolerable concentration of $0.2\mu\text{g/m}^3$ for long-term inhalation exposure to elemental mercury vapor and a tolerable intake of total mercury of $2\mu\text{g/kg}$ body weight per day (WHO, 2007).

The major sources of mercury emission are from natural, anthropogenic and re-emitted sources, whereas the most crucial anthropogenic sources are urban discharges, agriculture discharge, mining and combustion and industrial discharge. Combusted hydrocarbon fuels originates from petroleum and coal was also included as one of the major anthropogenic sources of mercury emissions to US according to US EPA 1997 (Diseases, 2008). Relatively, light hydrocarbon liquids which are crude oil, condensate, natural gas and the like produced from wells and exhaust combustion gas sometime contain mercury or mercury compound. Thus, this will contribute to several negative impacts on gas processing operation such as equipment degradation, toxic waste generation, increase risk to the health and safety of workers and poisoning of catalyst (Wilhelm & Bloom, 2000)

Mercury removal sorbent beds are used to scavenge the mercury from gas and liquid hydrocarbon streams. Several commercial processes are available for removing mercury and mercury compound from hydrocarbon but selection of the most effective system must be predicated since some of the mercury removal systems are targeted at gas phase treatment and some are targeted at liquids. Gas phase treatment systems primarily consist of sulfur impregnated carbon (U.S. Patent 8,598,072 B2), metal sulfide on carbon or alumina and regenerative molecular sieve. As for hydrocarbon liquid streams, the systems consist of iodide-impregnated carbon, metal sulfide on carbon or alumina, a mol sieve amalgam system and a two-step processing consisting of a hydrogenation conversion catalyst followed by metal sulfide reaction with elemental mercury. All the commercialized methods have both advantages and disadvantages that depend on feed composition and stream location.

In recent years, molecular imprinting has attracted considerable interest in many areas of chemistry, biochemistry and biotechnology owing to their high degree of selectivity and affinity towards the target molecule. Molecular imprinted polymers (MIPs)

applications including biosensor and chemosensor, microreactor, solid phase extraction (SPE), affinity chromatography and catalysis. The latest development in the technique of molecular imprinting have made an available polymers that can be used in the detection of drugs, toxins, pesticides, food components and other molecules that would be difficult to isolate otherwise.

MIPs were described as artificial synthesized macromolecular materials with prearrangement of structure and specific molecular recognition ability. This unique ability can recognize the template molecule used in the imprinting process even in the presence of compounds that having similar structure and functionality to the template. The MIPs tend to be simple and inexpensive to prepare and are generally rather robust in nature. MIPs also offer several advantages including the ability to tolerate high pressures, organic solvents, pH extremes and elevated temperature. Due to high degree cross-linking of MIPs, it provides distinctive chemical, mechanical and thermal long stability compared with their biological counterparts (Koesdjojo et al., 2007).

MIPs are generally prepared by involving the complexation in solution of a template molecule with functional monomers through covalent bonds followed by polymerization of these monomers around the template with the help of a cross-linker in the presence of an initiator. After the polymerization process, the template molecule are removed by extensive washing steps to disrupt the interactions between the template and the monomers thus leaving the cavities of binding sites that are complementary to the template in size, shape and position of the functional group (Pichon & Chapuis-Hugon, 2008). Hence, the obtained cavities can work as selective binding sites for the template molecule.

MIPs can be synthesized by three different imprinting approaches which are covalent, non-covalent and semi-covalent. Non-covalent approach become the most applied technique compared to the other approaches due to its generality and simplicity during the process of template removal and the resulting greater numbers of higher affinity sites. As for covalent route, it lack of general applicability due to difficulty in finding a suitable monomer to conjugate to the template. Non-covalent imprinting involving self-assembly of the template with functional monomer prior to polymerization, free radical polymerization with the cross-linking monomer and then template extraction followed by rebinding via non-covalent interaction such as hydrogen bonds, ion pairs, dipole-dipole interaction and Van der Waals forces (Caro et al., 2002).

1.2 Statement of Problem

The toxic effects of mercury have been observed for centuries. Mercury has been classified as one of the top ten hazardous heavy metals according to World Health Organization (WHO). This is because of its reactivity, extreme volatility and its relative solubility in water and living tissues. The detection of mercury has long held the intention of analytical community and as such, a large number of protocols have arisen. One of the major anthropogenic sources is from combusted hydrocarbon that originates from petroleum oil. The concentration of mercury in crude oil and natural gas is highly

dependent on geologic location and varies between approximately 0.01 ppb and 10 ppm. Although the total amount of mercury and mercury compound is considered low, but it give detrimental impacts on petroleum process. In addition, water streams that are by-products of equipment cleaning and water condensed from glycol regeneration can contain high concentrations of mercury that will cause aquatic contamination if severe treatment does not take place.

Various commercial methods are available in industries for mercury removal systems for hydrocarbon streams such as sulfur-impregnated carbon, metal sulfide on carbon or alumina, zeolite, activated carbon and so on. However, there are several disadvantages of this commercialized method for examples mol-sieve amalgamation sorbents do not operate at high efficiencies if organic forms of mercury are present in significant concentration. Other than that, sulfur-impregnated carbons are soluble in liquid hydrocarbon and cannot be used in process locations. For these reason, the sorbents that have ability to tolerate high pressures, organic solvents and provide high chemical and mechanical stability is still essential to be further investigates.

The aim of this research is to prepare molecular imprinted polymer (MIP) which can be used for the selective removal of mercury in mercury removal system. In this research, the method of initiation use will be free radical or chain growth polymerization. It is the most important method available today for the conversion of monomer into polymer and is exploited widely in industries. The parameter studies including pH studies, dosage studies, sorption kinetics, sorption isotherms, selectivity and reusability studies. Considering the versatility and high level selectivity and recognition that can be achieved, the future use of MIPs as a sorbents in mercury removal system is very promising.

1.3 Objectives:

The objectives of this study are:

1. To synthesize molecularly imprinted polymer (MIP) for the removal of mercury(II) ions.
2. To distinguish the MIP and NIP by spectroscopy analysis, morphological characterization, thermal studies and surface area and porosity analysis.
3. To study the binding capacity of mercury(II) ions toward MIP based on pH, kinetic, isotherm, selectivity and reusability.
4. To investigate the removal of mercury(II) ions from actual samples; crude oil and condensate.

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