UNIVERSITI PUTRA MALAYSIA

DISTRIBUTION AND SOURCE OF HYDROCARBONS IN THE SEDIMENTS OF THE SELANGOR RIVER, MALAYSIA

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By

NAJAT ABDULLAH EBRAHIM MASOOD

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for Degree of Doctor of Philosophy

July 2017

DEDICATION

In the name of Allah, the Beneficent, the Merciful

To my beloved husband and my daughters

To the memory of my late father

To my beloved mother and my dear siblings

In dedicate this dissertation

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement of the degree of Doctor of Philosophy

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Chairman : Normala bt. Halimoon, PhD
Faculty : Environmental Studies

Environmental pollutants such as anthropogenic hydrocarbons (AHs) are known to result from increased urbanization and act as indicators of anthropogenic activity. Hydrocarbons contamination is a great global concern due to their negative effects on human health and environmental consequences, such as toxic, mutagenic, carcinogenic, teratogenic and hepatotoxic effects. Consequently, a clear understanding and monitoring of distribution, sources, pathways and fate in the environment and ecosystems of AHs is of high importance in sediment samples collected from Kuala Selangor River, which is located on the west coast of Peninsular Malaysia, in order to determine the distributions and sources of hopanes, alkanes, polycyclic aromatic hydrocarbons (PAHs), and linear alkylbenzenes (LABs) in four periods. The sediment samples were collected from the river and its estuary during the rainy inter-monsoonal period, the dry inter-monsoonal period, the North-East Monsoon (NEM) period, and the South-West Monsoon (SWM) period because during these periods the samples are expected to contain varying amounts of pollutants. The collected samples were homogenized, freeze-dried, extracted, cleanup, fractionated and analyzed using gas chromatography mass spectrometry. The results of this study revealed that the concentrations of PAHs for the 13 sampling sites pooled over the four studied periods ranged from 341.33 ng g^{-1} dw (in the SWM period) to 651.64 ng g^{-1} dw (in the wet inter-monsoon period) with the mean value of 471.05 ng g^{-1} dw. In other respects, the outcomes of the one-way ANOVA indicated that the differences between the PAHs among the sampling stations were significant ($p < 0.05$). The diagnostic ratios of individual PAHs in sediments indicated both petrogenic and pyrogenic origin PAHs with significant dominance of pyrogenic source. The comparison of PAHs with Sediment Quality Guidelines (SQGs) indicates that the levels of the PAHs in the surface sediments of Selangor River are unlikely to cause any adverse biological effects to the aquatic organisms, including those that are closely associated with sediments. No significant correlation was found between AHs and organic carbon, which means that the distribution of AHs was not affected by the organic carbon content. In the four studied periods, the concentrations of the LABs in Selangor River ranged from 23.70 to 113.30 ng g^{-1} dw with an arithmetical mean of 54.04 ± 22.34 ng

g^{-1} dw. The average concentration of the total LABs was found to be higher in the wet season (65.20 ± 25.04 ng g^{-1} dw) than in the other seasons. The average values of the ratio of long-chain to short-chain LAB (L/S), C_{13}/C_{12}, and the ratio of Internal to External isomers (I/E) in the sediments of Selangor River during the four studied periods were 2.03 ± 1.06, 1.74 ± 0.28, and 0.6 ± 0.17, respectively, indicating inputs of LABs from direct wastewater discharges. The I/E ratio decreased from upstream to downstream of Selangor River. This indicates that the sedimentary LABs downstream of the river (estuary) were more degraded than those in the upstream. The concentrations of total alkanes (nC_{10}-nC_{36}) ranged from 967 to 3,711 µg g^{-1} dw in sediment samples. In addition, the results of this study indicated that the alkanes in the sediment samples originated from diverse sources. Fresh oil, terrestrial plants (riverine area), and heavy/degraded oil (estuarine area) were the predominant source of alkanes in sediments. The alkanes originating from higher plants were mostly predominant in sediments from the stations located in upper parts of the river. The levels of hopanes ranged from 235 to 1,044 ng g^{-1} dw in sediments. The C_{29}/C_{30} hopanes ratios were similar with those of used crankcase oil and Middle East Crude Oil (MECO), suggesting MECO as a major source of petroleum hydrocarbons (PHCs) in the sediments of Selangor River. In sum, presence of selected hydrocarbons (PAHs, LABs, alkanes, and hopanes) in the sediments of Selangor River, Malaysia, was studied during four periods. The results show that the agricultural, urban, and industrial activities in the state of Selangor, coupled with a high population growth, have caused deterioration in the quality of its river water. The concentrations of the investigated hydrocarbons varied from sampling site to another and from one period to another. Further studies should be conducted to address this problem.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

TABURAN DAN SUMBER HYDROCARBON DALAM PERMUKAAN SEDIMEN SUNGAI SELANGOR, MALAYSIA

Oleh

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Pencemar alam sekitar seperti antropogenik hidrokarbon (AHs), diketahui berpunca akibat daripada peningkatan perbandaran dan bertindak sebagai petunjuk aktiviti antropogenik. Pencemaran hidrokarbon menjadi kebimbangan global yang besar disebabkan oleh kesan negatifnya terhadap kesihatan manusia dan alam sekitar, seperti kesan toksik, mutagenik, karsinogenik, teratogenik dan hepatoksik. Oleh itu, pemahaman dan pemantauan yang jelas mengenai taburan, sumber, laluan dan nasib dalam alam sekitar dan ekosistem AHs adalah sangat penting di dalam sampel sedimen yang diambil dari Sungai Kuala Selangor, yang terletak di pantai barat Semenanjung Malaysia, dalam menentukan taburan dan sumber hopan, alkana, hidrokarbon aromatik polisiklik (PAHs), dan alkilbenzena linear (LABs) dalam empat tempoh. Sampel sedimen diambil dari sungai dan muara sungai tersebut ketika tempoh peralihan monsun hujan, tempoh peralihan monsun kering, tempoh monsun timur laut (NEM), dan tempoh monsun barat daya (SWM) kerana pada waktu tersebut, sampel dijangkakan mengandungi pelbagai bahan pencemar. Sampel yang diambil telah dihomogenkan, dibekukeringkan, diekstrak, dibersihkan, difraksinasi dan dianalisa menggunakan kromatografi gas spektrometer jisim. Keputusan kajian ini menunjukkan bahawa kepekatan PAHs yang diperoleh dari 13 kawasan persampelan untuk empat tempoh kajian berjulat antara 341.33 ng g^{-1} dw (dalam tempoh SWM) hingga 651.64 ng g^{-1} dw (dalam tempoh basah intermonsun) dengan nilai purata 471.05 ng g^{-1} dw. Dalam aspek lain pula, hasil daripada ANOVA sehala menunjukkan bahawa perbezaan PAHs antara setiap stesen persampelan adalah signifikan ($p < 0.05$). Nisbah diagnostik PAHs secara individu dalam sedimen menunjukkan kedua-dua PAHs adalah petrogenik dan pirogenik dengan sumber pirogenik yang paling ketara. Perbandingan PAHs dengan panduan kualiti sedimen (SQGs) menunjukkan paras PAHs di permukaan sedimen Sungai Selangor bermungkinan rendah untuk memberikan kesan biologi ketara pada organisma akuatik, termasuklah yang berkaitan dengan sedimen. Tiada hubungan signifikan antara pengagihan AHs dan karbon organik di mana, taburan AHs tidak terkesan dengan kandungan karbon organik. Dalam keempat-empat tempoh kajian, kepekatan LABs di Sungai Selangor berjulat antara 23.70 ke 113.30 ng g^{-1} dw dengan purata aritmetik 54.04 ± 22.34 ng g^{-1} dw. Kepekatan purata LABs didapati lebih tinggi pada musim hujan (65.20 ± 25.04 ng g^{-1} dw) berbanding musim-musim lain.

Nilai purata nisbah rantai panjang terhadap rantai pendek LAB (L/S), C_{13}/C_{12}, dan nisbah dalaman untuk isomer dalaman terhadap isomer luaran (I/E) dalam sedimen Sungai Selangor untuk keempat-empat tempoh kajian adalah 2.03 ± 1.06, 1.74 ± 0.28 dan 0.6 ± 0.17 menunjukkan kehadiran LABs adalah berasal secara terus daripada pelepasan air kumbahan. Nisbah I/E menunjukkan penurunan dari hulu ke hilir Sungai Selangor. Ini menunjukkan bahawa sedimen LABs di kawasan hilir sungai (muara) lebih terurai daripada hulu sungai. Kepekatan jumlah alkana (nC_{10}-nC_{36}) adalah berjulat antara 967 ke 3,711 μg g^{-1} dw dalam sampel-sampel sedimen. Tambahan pula, keputusan kajian ini menunjukkan bahawa alkana dalam sampel sedimen berasal dari pelbagai sumber. Minyak segar, tumbuhan daratan (kawasan sungai) dan minyak berat/terurai (kawasan muara) merupakan punca utama alkana di dalam sedimen. Alkana yang berasal daripada tumbuh-tumbuhan komplek adalah paling ketara di dalam sedimen stesen yang terletak di hulu sungai. Paras hopana dalam sedimen pula adalah antara 235 hingga 1,044 ng g^{-1} dw dalam sedimen. Nisbah C_{29}/C_{30} hopana adalah sama dengan nisbah bagi minyak crankcase dan Minyak Mentah Timur Tengah (MECO), mencadangkan bahawa MECO menjadi sumber utama hidrokarbon petroleum (PHCs) dalam sedimen Sungai Selangor. Keseluruhannya, kehadiran hidrokarbon terpilih (PAHs, LABs, alkana, dan hopana) dalam sedimen Sungai Selangor, Malaysia, telah dikaji sepanjang empat tempoh. Hasil kajian menunjukkan bahawa aktiviti-aktiviti pertanian, perbandaran dan perindustrian di negeri Selangor, ditambah pula dengan pertumbuhan penduduk yang tinggi, telah menyebabkan kemerosotan kualiti air sungai tersebut. Kepekatan hidrokarbon adalah berbeza di antara setiap lokasi dan tempoh persampelan kajian selanjutnya perlu dijalankan untuk menangani masalah ini.

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I certify that a Thesis Examination Committee has met on 13 July 2017 to conduct the final examination of Najat Abdullah Ebrahim Masood on her thesis entitled "Distribution and Source of Hydrocarbons in the Sediments of the Selangor River, Malaysia" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ACL	Average chain length
Ant/(Ant+Phe)	Ratio of anthracene to phenanthrene plus anthracene
BaA/(BaA+Chr)	Ratio of Benzo(a)anthracene to benzo(a)anthracene plus chrysene
C_{13}/C_{12}	The ratio of ΣC13 - LAB/ΣC12 - LAB
C_{17}/pristane	Ratio of C_{17} n-alkane to pristane
C_{18}/phytane	Ratio of C_{18} n-alkane to pristane
C_{29}/C_{30}	Ratio of 17α, 21β (H)-30-norhopane to17α, 21β (H)-hopane
ΣC_{31}-C_{35}/C_{30}	Ratio of sum of 17α, 21β (H)-C_{31} homohopane to 17α, 21β (H)-C_{35} homohopane relative to 17α, 21β (H)-hopane
∑cPAHs	Sum of carcinogenic PAHs
CPI_s	Carbon preference indices
DCM	Dichloromethane
D(%)	The average degradation
dw	Dry weight
ERL	Effects range low
ERM	Effect range median
eV	Electron Volt
Fluo/(Fluo +Pyr)	Ratio of fluoranthene to fluoranthene plus pyrene
Fluo/Pyr	Ratio of fluoranthene to pyrene
GC-MS	Gas Chromatography-Mass Spectrometry
HMW	High Molecular Weight
I/E	The ratio of Internal to External isomers
IIS	Internal Injection Standard
K_{ow}	Octanol-water partitioning coefficient
LABs	Linear alkylbenzenes
LMW	Low Molecular Weight
LMW/HMW	Ratio of Low Molecular Weight to High Molecular Weight isomers
InP/(InP+BgP)	Ratio of indeno(1,2,3-cd)pyrene to indeno(1,2,3-cd)pyrene plus benzo(g,h,i)perylene
L/S	The ratio of long-chain to short-chain LAB
MECO	Middle East Crude Oil
MH	Major hydrocarbon
MP/P	Ratio of methylphenanthrenes to phenanthrene
m/z	Mass to charge ratio
alkanes	Normal alkanes
Na_2SO_4	Sodium sulfate
nd	Not detected
PAHs	Polycyclic aromatic hydrocarbons

PC	Principal component
PCA	Principal Component Analysis
PEL	Probable effects level
PHC	Petroleum hydrocarbon
Phe/Ant	Ratio of phenanthrene to anthracene
Phy	Phytane
Pri	Pristane
Pr/Ph	Ratio of pristane to phytane
S	Station
SEACO	South East Asia Crude Oil
SIM	Selected Ion Monitoring
SIS	Surrogate Internal Standard
SLC	Screening level contamination
SQGs	Sediment quality guidelines
TARs	Terrigenous/aquatic ratios
TEF	Toxicity equivalency factor
TEL	Threshold effect level
TEQ	Toxicity equivalency quotient
TOC	Total organic carbon
T_s	18α(H)-22,29,30-trisnorneohopane
T_m	17α(H)-22,29,30-trisnorhopane
T_m/T_s	Ratio of 17α-22,29,30-trisnorhopane to 18α-22,29,30-trisnorhopane
UCM	Unresolved complex mixture
USEPA	United States Environmental Protection Agency

CHAPTER 1

INTRODUCTION

1.1 Background of the study

The eclectic integration of the economic, social, and cultural aspects has led to a rapid physical growth in the urban areas of Malaysia. Due to these rapid development activities, several environmental problems, including pollution, have been created. As a matter of fact, human activities such as lodging and waste generation and improper discharge can harmfully contribute to the environmental degradation problems. The various human activities have serious environmental complications and may overwhelm the surroundings if there are no instantaneous precautionary steps taken by all stakeholders.

Selangor is one of the 13 states of Malaysia and one of the states lying on the west coast of Peninsular Malaysia. Its geographical position, in the core of Peninsular Malaysia, has contributed to rapid development of the state. Hence, the State of Selangor is considered as the transportation and industrial hub of Malaysia which, in turn, attracts both interior migrants such as those coming from the other Malaysian States and exterior tourists coming from different countries (Alsalahi et al., 2014). The basin of Selangor River faces serious environmental degradation from urbanization, industrialization, and population growth. Currently, the major land-based pollution sources in the basin are (Ong, 2005), poorly-treated or untreated domestic sewage, siltation from land clearing, construction and sand mining activities, run-off from agricultural land, and untreated effluent from industry. In the past few decades, the anthropogenic activities associated with rapid industrialization and urbanization growth like fossil fuel combustion, discharge of domestic sewage, and incineration of industrial waste contributed greatly to pollution, especially in the marine and coastal environments in Malaysia. Among the pollutants, hydrocarbons are among the most threatening pollutants which have been widely studied. The Strait of Malacca is one of the world's busiest supertanker routes (Zakaria et al., 2001). It is surrounded by an area which is rich with natural resources that include oil and natural gas. Therefore, there is a variety of potential sources of petroleum in these coastal areas. Moreover, the coasts of Malaysia are subjected to various potential sources of petroleum pollution, including routine and accidental oil spills from tankers, spillage of crude oils from inland and offshore oil fields, and run-off from urban lands.

Because of their seriousness and being among the most widespread organic pollutants, the polycyclic aromatic hydrocarbons (PAHs) are given more attention than other pollutants. Pollution with PAHs is one of the primary environmental problems facing humanity at present, mainly owing to the fact that PAHs are toxic, mutagenic, and carcinogenic to humans and other organisms. Additionally, they are subjected to bioaccumulation and concentration in the aquatic food web. Furthermore, they are

relatively persistent in the environment. Thus, the focus of the current study is investigation of the concentrations and the origins of four groups of hydrocarbons, namely, alkanes, hopanes, linear alkylbenzenes (LABs), and PAHs in the sediments of Selangor River, which faces the Strait of Malacca. The results of this study give an indication of the magnitude of pollution of Selangor River with these pollutants and the distribution of these pollutants along the river. Studying the levels and distribution of the PAHs and LABs in the sediments is highly important as both groups of compounds are highly hydrophobic and have large values of the octanol-water partition co-efficient (K_{ow}). As such, they are expected to partition and accumulate into the sediments and the lipid parts of biota. Besides, some PAHs are carcinogenic and mutagenic in nature. Accordingly, the identification and quantification of PAHs in sediments are significant in order to mark clean-up areas.

1.2 Problem statement

The aquatic and terrestrial ecosystems are exposed to the threat of hydrocarbon accumulation as a result of the increase in human activities along the coastline facing the Strait of Malacca. This has led to pollution of the marine and coastal environments, including the mangrove forests, rivers such as Selangor River. More than 80% of the hydrocarbons (HCs) found in the environment are produced by anthropogenic activities while the other 20% are released naturally through oil seeps and biogenic sources. The major HCs of environmental prevalence and, hence, concern are the straight chain alkanes, cycloalkanes, and aromatics. Relatively, the HCs are of the greatest concern, which is an issue that is related to the ubiquity of the PAHs in the environment. The PAHs are considered as one of the most important classes of micro-organic pollutants, produced mostly via incomplete combustion of organic materials containing carbon, hydrogen, and other minor elements (Bakhtiari et al., 2010). In this study, the PAHs were selected because they are persistent, toxic, mutagenic, and carcinogenic and because they have teratogenic and hepatotoxic effects (Cao et al., 2009; Karami et al., 2012a; Masiol et al., 2012). These pollutants have worrying human health implications and are also of concern for potential adverse effects on a range of organisms. They have not only affected biological changes in, and sometimes death of, aquatic and terrestrial ecosystems, but also the tourism industry, which is one of the main economic engines of Malaysia that has experienced damages and loss. Therefore, a study on hydrocarbons contamination in sediments is required in order to determine the current level of hydrocarbons in Peninsular Malaysia especially the sediment regions located along the Straits of Malacca such as the chosen site, the Selangor River.

1.3 Significance of the study

Study on hydrocarbons in sediments will significantly provide the scientific community and local environmental managers with useful data on the various characteristics of hydrocarbons in the aquatic environment of Selangor River that can be utilized by decision makers in the future planning for the purpose of achievement of sustainable development. The current study is important for scientists, environmentalists, educators, and the tourism managers since it will contribute to further understanding of the effects of the human activities and the human by products on the ecosystems of

Selangor River. It is expected that the study results can help understand the current condition of the hydrocarbons pollution, project the future outcomes of its anticipated effects, and change certain human activities or impose certain regulations to save the aquatic and terrestrial environment. Furthermore, none of the previous studies employed source apportionment techniques for the identification and quantitative evaluation of hydrocarbons sources in Selangor River. Therefore, in view of the pollution condition and the importance of the investigated river as source of food and drinking water, the characterization of hydrocarbons and their sources by apportionment techniques is considered as a novel approach that can contribute substantially to the understanding of the primary source(s) of hydrocarbons in sediments. The results of this study will be compared with the proposed PAH levels of sediment quality guidelines in order to evaluate the biological effects of sedimentary PAHs to the aquatic organisms, especially the ones that are closely associated with sediment. Finally, a number of new findings on PAHs fate in Malaysian sediment will be scientifically shown through this study.

1.4 Research questions

Whatever the origin of contamination, benthic habitats are preferential environmental sink for many contaminants, since many pollutants originally introduced into the water column have affinities for sediment particles. Because sediments are also an important biological habitat, incorporation of toxicants into the food web is influenced by toxicant concentrations in sediments, which represent the main sink for PAHs and constitute PAH reservoirs. Therefore, this study was initiated to address the following four research questions:

1. What the compositional patterns and sources of alkanes, hopanes, LABs and PAHs in the sediments of Selangor River in the four studied periods?
2. Are there significant differences in hydrocarbon concentrations among the four periods under study?
3. Are there significant relationships between total organic carbon (TOC) and the concentrations of alkanes, hopanes, LABs and PAHs in the surface sediments in the study area?
4. Can LABs serve as molecular markers for identification of the sources of PAHs in sediments? and is there a toxic effect of PAHs on human health and ecological environment.

1.5 Scope of the study

This research has been conducted on the sediments of Selangor River to investigate the level of hydrocarbon pollution in these sediments. Besides, the study aimed at differentiating hydrocarbon contamination, linking the molecular markers to their sources. In other words, the purpose of this study was to apply environmental fingerprinting techniques in order to determine the levels, origins, and distributions of alkanes, hopanes, LABs and PAHs in the sediments of Selangor River in four periods. Therefore, this study adopts the principles of environmental forensics, including source

apportionment of compounds in environmental samples to Anthropogenic Waste Indicators (AWI).

The AWIs are indicators of man-made wastes that consist of chemical compounds originating from pharmaceuticals, detergent disinfectants, fragrances, antimicrobials, PHC, pesticides, and other waste-borne materials present in wastewater and agricultural and urban run-off, which are commonly referred to as AWI compounds. The AWIs include endocrine-disrupting compounds (EDCs) that have detrimental reproductive effects on fish (Brian et al., 2007) and humans (Guillette, 1995). The AWI can also be utilized to indicate other anthropogenic pollutants like PAHs and LABs. Selangor River was chosen because the river flows through the commercial, industrial, and residential areas facing the Strait of Malacca. Furthermore, urban and other development areas surround the river.

1.6 Objectives of the study

The main and the leading objective of this study was to analyze the degree of anthropogenic pollution and used of hydrocarbon markers to investigate the distribution of anthropogenic inputs to the sediments of Selangor River, Malaysia. However, under the main objective of the study, the following four sub-objectives are listed:

1. To assess the compositional patterns and sources of alkanes, hopanes, LABs and PAHs in the sediments of Selangor River in four periods: the dry inter-monsoonal period, the wet inter-monsoonal period, the south-west (SW) monsoon, and the north-east (NE) monsoon periods.
2. To compare the concentrations of the alkanes, hopanes, LABs and PAHs in different sampling stations during the four study periods through the use of multivariate data analysis.
3. To determine the relationships between total organic carbon (TOC) and the concentrations of alkanes, hopanes, LABs and PAHs in the surface sediments in the study area.
4. To investigate the utility of LABs as molecular marker for identification of the sources of PAHs in sediments and to evaluate the PAHs potent toxicity on human health and ecological environment.

1.7 Organization of the thesis

The outline presented next describes the different chapters of this thesis and assists the reader in understanding the flow of its chapters and main sections.
The thesis consists of five chapters. Chapter 1 defines the background and specifies the purpose of this investigation.

Next, Chapter 2 reviews and discusses the literature relevant to the study. It highlights the characteristics of alkanes, hopanes, LABs and PAHs in the light of their sources

and uses. The chapter also spotlights the relevant chemical and environmental properties of alkanes, hopanes, LABs and PAHs, as well as the main risks of human exposure to PAHs. It also provides international and national standards with regard to PAHs. Finally, this Chapter discusses trends in alkanes, hopanes, LABs and PAHs, and sheds some light on the shortcomings of the related research.

Then, Chapter 3 describes the methodology and experimental procedures followed in the study. It describes the sampling and the data collection procedures.

Afterwards, Chapter 4 presents a detailed analysis of the data collected and provides illustrations along with the discussion of the results. It also compares the results of this study with those of previous studies.

Finally, Chapter 5 concludes the thesis. It presents a summary of the main research findings, the major conclusions, and some recommendations for future research.

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