UNIVERSITI PUTRA MALAYSIA

SOURCE IDENTIFICATION AND DISTRIBUTION OF PETROLEUM HYDROCARBONS IN SOIL AND SEDIMENTS OF THE NORTHERN PERSIAN GULF COAST

FATEMEH VALIZADEHKAKHKI

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SOURCE IDENTIFICATION AND DISTRIBUTION OF PETROLEUM HYDROCARBONS IN SOIL AND SEDIMENTS OF THE NORTHERN PERSIAN GULF COAST

By

FATEMEH VALIZADEHKAKHKI

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

May 2013

DEDICATED

To my Mother

To my Father and my Sisters

To my Husband

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

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May 2013

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The purpose of this study is to use environmental fingerprinting technique and to identify oil spills and their relationship with the oil derived from known oil fields. This is the first comprehensive study on PAHs and *n*-alkanes distribution as oil spill markers in the Persian Gulf to investigate petroleum contamination in soil and sediment in the three regions identified as sources of oil and gas. Soil and sediment from three provinces in the northern Gulf has been selected. Soil samples (0-10 cm) were collected from rural land, industrial, urban, and agriculture. Sediment samples were collected from intertidal area, 3 offshore and 2 rivers samples were collected. Intertidal sediment was collected using stainless steel spoon from 5 top cm. Offshore sediment samples were collected using VanVeen grab from 10 km into the Gulf. The results indicated that Total PAHs range between 42.76 to 5596.4 $ng.g^{-1}$ for rural soils, 460 to 1730.4 $ng.g^{-1}$ for industrial soil, 165.6 to 3442.3 $ng.g^{-1}$ for urban soil and 57.3 to 3633.5 $ng.g^{-1}$ for agricultural soils. Polynuclear aromatic hydrocarbons in rural area showed higher

concentration than other stations and it is associated with petrogenic input as these sites are located near oil fields and oil transferring pipelines. In addition, the concentrations for sediment showed that TPH is from 184.5 to 2771.8 $ng.g^{-1}$ dry weight. Analysis of the results and application of biomarker ratios such as MP/P ratio showed that the main source of PAHs input is petrogenic sources. Consequently, most of the pollution in the sampling region belonged to oil spill, crude oil and petroleum products. On the other hand, analysis of the *n*-alkanes revealed there was principally higher molecular weight and even carbon numbers. Therefore, it indicates that the major contribution of the *n*-alkanes are related to crude oil and petroleum products and it is not derived from biogenic sources as the number of odd carbon is very low. Additionally, high vanadium concentration reflects an oil spill background to the area, since vanadium is the major heavy metal constituent of crude oil. To conclude, analysis of all components illustrated that contamination in the mentioned region is derived from direct inputs of petroleum products and crude oil to the land and Gulf. Hence, monitoring and long-term investigation to produce a reference profile is a practical tool to verify the contribution of anthropogenic compounds to this environment.

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

PENGENALAN SUMBER DAN TABURAN HIDROKARBON PETROLEUM DI DALAM TANAH DAN SEDIMEN PANTAI UTARA TELUK PARSI

Oleh

FATEMEH VALIZADEHKAKHKI

May 2013

Pengerusi: Professor Mohammad Pauzi Zakaria, PhD

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Tujuan kajian ini dijakalan adalah bagi mengenal pasti tumpahan minyak dengan merggunakan telunjuk cap jari alam sekitar dan menguji hubungannya dengan minyak yang diperolehi daripada telaga minyak yang diketahui. Ini adalah kajian menyeluruh yang pertama bagi PAH dan n-alkana pengedaran sebagai penanda tumpahan minyak di Teluk Parsi untuk menyiasat pencemaran petroleum dalam tanah dan sedimen di tiga kawasan yang dikenal pasti sebagai sumber utama minyak dan gas. Tanah dan sedimen dari tiga wilayah di Teluk utara telah dipilih. Sampel tanah (0-10 sm) telah diambil dari pada tanah luar bandar, industri, bandar, dan pertanian. Sampel sedimen telah diambil di kawasan pasang surut, 3 luar pesisir dan 2 sampel sungai telah dikumpulkan. Sedimen pasang surut telah dikumpulkan menggunakan sudu keluli tahan karat daripada 5 sm atas. Sampel luar pesisir telah dikumpulkan menggunakan grab VanVeen dari 10 km ke Teluk. Keputusan menunjukkan bahawa pelbagai TPH antara 42.76-5596.4 $ng.g^{-1}$ untuk tanah luar bandar, 460-1730.4 $ng.g^{-1}$ untuk kegunaan industri, 165.6-3442.3 $ng.g^{-1}$ untuk bandar dan 57.3-3633.5 $ng.g^{-1}$ untuk tanah pertanian. hidrokarbon aromatik

polinkleor di kawasan luar bandar menunjukkan kepekatan yang lebih tinggi daripada stesen lain dan ia dikaitkan dengan input pembakaran tidak sempurna seperti laman-laman yang terletak berhampiran dangan kawasan minyak dan saluran paip memindahkan. Di samping itu, kepekatan sedimen menunjukkan bahawa TPH adalah 184.5-2771.8 $ng.g^{-1}$ dalan berat kering. Analisis keputusan dan permohonan nisbah penunjuh seperti nisbah MP/P menunjukkan bahawa sumber utama input PAH adalah sumber pembouaron tidak sempurna. Oleh itu, sebahagian besar daripada pencemaran adalah dari pada tumpahan minyak, minyak mentah dan produk petroleum. Sebaliknya, analisis n-alkana menunjuhan terdapat berat molekul yang lebih tinggi dan juga nombor karbon juga. Oleh itu, ia menunjukkan bahawa sumbangan utama n-alkana yang berkaitan dengan minyak mentah dan produk petroleum dan ia tidak berasal dari pada sumber-sumber biogenik kerana bilangan ganjil karbon adalah sangat rendah. Memandanguan, kepekatannya adalah sangat tinggi dan ia mencerminkan latar belakang tumpahan minyak ke kawasan itu. Vanadium adalah konstituen logam berat utama minyak mentah. konkklusinya, analisis semua komponen digambarkan bahawa pencemaran di rantau ini disebut berasal dari produk petroleum dan minyak mentah dari tanah dan Teluk. Oleh itu, pemantauan dan penyiasatan jangka panjang untuk menghasilkan profil rujukan adalah sangat praktikal untuk mengesahkan kesan sumbangan sebatian antropogenik kepada alam sekitar ini.

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I certify that a Thesis Examination Committee has met on 17 May 2013 to conduct the final examination of Fatemeh Valizadehkakhki on her thesis entitled "Source Identification and Distribution of Petroleum Hydrocarbons in Soil and Sediment of the Northern Coast of the Persian Gulf" in accordance with the Universities and University Colleges Act 1971 and 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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DECLARATION

I declare that the thesis is my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at University Putra Malaysia or at any other institution.

FATEMEH VALIZADEHKAKHKI

Date: 17 May 2013

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LIST OF ABBREVIATIONS

Nap	Naphthalene
DBT	Dibenzothiophene
Phe	Phenanthrene
Ant	Anthracene
2MA	2-methylanthracene
3MP	3-methylphananthrene
2MP	2-methylphananthrene
9MP	9-methylphananthrene
1MP	9-methylphananthrene
Flu	Fluoranthene
Pyr	Pyrene
1MPyr	1-methylpyrene
Chy	Chrysene
P	Perylene
BaA	Benzo (a) anthracene
BkF	Benzo (k) fluornthene
BeA	Benzo (e) acephenanthrene
BeP	Benzo (e) pyrene
BaP	Benzo (a) pyrene
DBahA	Dibenzo (a,h) anthracene
V	Vanadium
Ni	Nickel
Pr	Pristane
Ph	Phytane
DMBaAnt	Dimethyl benzo (a) anthracene
UCM	Unresolved Complex Mixture
CPI	Carbon Preference Index
PI	Pyrogenic Index
WI	Weathering Index

CHAPTER 1

INTRODUCTION

1.1 General Introduction

1.1.1 Oil Pollution in Persian Gulf

The Persian Gulf, located in southwestern Asia, is a vast expansion of the Indian Ocean situated between Iran and the Arabian Peninsula. The Persian Gulf and its coastal areas are the world's largest particular source of crude oil and related industries govern the region. Persian Gulf with its own uniqueness, geographical and ecological value of marine ecosystems is among the rarest maritime ecosystem. It is distinguished as a great economic source of oil and gas and two third of the oil resources of the world is to be found here. Each year around 25000 oil and non-oil tankers travel in it. So it is one of the world's busiest waterways for oil and non-oil tanker Khan (2002). Owing to the oil spill, ballast water, discharging, the platform exploitation of oil wells and accidents such as collisions, and the destruction of fire tankers and oil platforms, to the Persian Gulf, it is exceedingly contaminated Mohammed and Al-Ssadh (1996); Munawar *et al.* (2002); Law and Hii (2006). Due to the shallow depth, the hot weather, salty water, limited contact with the world free water and the immense issues of harvested energy sources, a unique ecosystem is produced in the Persian Gulf. This led to be vulnerable against the sources of pollution. Releasing of municipal waste to the Persian Gulf, introducing of hot sewage of the plants, discharge of saline waste water from fresh

water equipments, use of seabed sand for urban and industrial activities, the oil exploration and exploitation of the seabed, possible eruption of oil wells, tankers clash and sink, discharge of the balanced water of the ship, military attack to oil tankers oil fields and explosion of the oil in the uninterrupted wars of the region, caused to the Persian Gulf, a susceptible environment. About 50 percent of the pollution of the Persian Gulf caused by non-oil pollutants and 50 percent is from oil sources. So far possible sources of Petroleum pollution in Iran are Domestic oil production, Old transferring pipelines, Oil tanker sources and Industries.

1.2 Hydrocarbon Pollution in the Middle East

During the Iraq invasion to Kuwait the largest oil contamination occurred by burning of oil wells, from 4 to 6 million tons of oil fuel burned or released to the region daily. Contamination level of the Persian Gulf is stated 47 times higher than the average. It was said that oil contamination caused by Iraq invasion to Kuwait in 1991 will not be removed till 200 years. In the third decade of the war it was declared that demolish was more than World War II. Moving on to another part, in the direction of blasting of oil wells, wind transferred smokes, toxic gases, particles, and dust from Iraq to Iran. In the first place, the provinces which are mostly damaged are Khuzestan, Boushehr, and countries such as North Saudi Arabia, Bahrain, and Qatar. Secondly Ilam, Kohkyloyeh and Boyerahmad, Charmahal, South of Fars, which makes the soil contaminated and the first symptom in human body was diarrhea. Consequently around 18 percent of the soil was polluted. Moreover, by means of a population growth rate of about 3% per year,

environmental problems are probable to become more significant in the future, unless measures are taken to ameliorate environmental and natural resource management.

1.3 Chemical Compositions of Crude Oil in Middle East

Crude oil compositions vary widely depending on the sources of carbon from which the oils are generated, the geologic environment in which they migrated, and from which reservoir (such as Middle East or North Sea), they may have dramatically varied compositions in the C_5-C_{44} carbon range, such as relative amounts of paraffinic, aromatic, and asphaltenic compounds; large differences in the *n*-alkanes, isoprenoids, and cyclic-alkanes (such as alkyl cyclo-hexanes) concentrations and distribution patterns and Unresolved Conplex Mixture (UCM) profiles. The most prominent aliphatics in most crude oil are the normal (straight chain) alkanes. In general, most crude oils exhibit an *n*-alkanes distribution profile (GC-FID and GC/MS at m/z 85) of decreasing abundances with increasing carbon number. The maximum *n*-alkanes within the profile are variable from oil to oil. The smoothness of the *n*-alkanes distribution profile in crude oil can be diagnostic.

1.4 Scope of the Research

To realize the fate and behavior of spilled oil in the environment, to differentiate spilled oils and to link the molecular markers to the original sources and to assess the environmental damage and prediction of the possible long-term impact of spilled oils on

the environment this research has been conducted to carry out within the northern coasts of Persian Gulf, a distinguished location of oil and gas reservoirs.

The purpose of this study is to apply environmental fingerprinting techniques and to identify oil spills and their relationship with oils originating from known oil fields. This study also focused on the polycyclic aromatic hydrocarbons and *n*-alkanes contamination, in the northern coasts of Persian Gulf in order to study petroleum pollution in soils and sediments in three provinces being identified as the sources of oil and gas fields.

Despite the theory proven for the contaminated regions by crude oil and petroleum products, nearly most of the sites especially for the agricultural lands and a major river in Bushehr Province did not show a dramatic impact of oil contamination and their contamination was less than the expectation compared to the other reported contamination around the world. Surprisingly, most of the sites showed a chronic effect of the contamination except for one site in Bushehr Province which was contaminated around a year ago.

1.5 Problem Statements

Persian Gulf with its unique and strategic situation is one of the major regions to produce oil, gas and their products and derivatives. Since there are numerous exploration and exploitation of oil from inland and offshore stations, therefore, it is necessary to investigate the effects of the exploitation on the environment. Crude oil

and petroleum products are the major sources of PAHs into the environment. Moreover, after Iraqi troops withdrew from Kuwait at the end of the Persian Gulf War in 1991, they set fire to over 600 oil wells and pools of spilled oil in Kuwait. It causes the region to be unwanted polluted by pyrolitic PAHs emitted from the great flames of the oil wells. It is obviously deposited in the environment around the region and specifically in the northern coasts of Persian Gulf being located in Iran. Once, this contamination introduced through the region it influenced on marine organisms, water, sediment and soil. It affects on human life as well since some of them are highly toxic and some of them are carcinogenic. Mostly 4, 5 and 6 ringed PAHs are considered carcinogenic based on EPA investigation In order to control the region some background data are needed. Primarily this study produced some background data and based on them control and remediation of the contaminated area will be performed. There are some gaps on the information about pollution related to this region and where this pollution refer back is not clear. Therefore, this study conducted to get the total concentrations of the PAHs and individual components and secondly to indicated that they belong to what type of sources.

1.6 The Principal Gas Reservoir in the Persian Gulf

The issue of petroleum pollution has been a controversial and much disputed subject so attentions to the area producing crude oil and gas are noticeable. One of the most disputed regions in the south of Iran is Asaluyeh being selected as the site of the Pars Special Energy Economic Zone (PSEEZ) facilities in Bushehr province. Owing to, it is the nearest land point to the largest natural gas field in the world, the South Pars/North

Dome Gas-Condensate field. The PSEEZ as it is known has been allocated 100 square kilometers of land at Asaluyeh for a variety of complexes and services. The site is a compilation of different refineries and plants (identified as phases). A sum of 27 phases are envisaged (12 gas, 15 petrochemical), in addition to a mix of light and heavy industry. Moreover, 10 refineries and 7 petrochemical complexes were already prepared in 2009. Each of the phases of the South Pars project is expected to have an average wealth spend of approximately US$1.5bn, and most will be led by foreign oil firms running in partnership with local companies Maleki (2007). The different plants and complexes presently run for around 12 km, and more are being constructed. A series of gas flares which line the facility are directly apparent, including one enormous flare principally, with flames of almost 100 m in height. This flare is visible far out to the sea. The field is the prevalent gas field in the world, shared between Iran and Qatar, which contains 1,800,000,000,000,000 cubic feet (51,000 km^3) gas in place. The South Pars Field is the name of northern part, which is positioned in Iranian waters and the North Dome is the name of southern part, which is situated in Qatari waters. South Pars Field was discovered in 1990 by NIOC (National Iranian Oil Company). Iranian sections contain 500,000,000,000,000 cubic feet (14,000 km^3) of gas in place and around 360,000,000,000,000 cubic feet (10,000 km^3) of recoverable gas (National Iranian Petrochemical Company and Special Economic Zones in Iran). It covers an area of 500 square miles (1,300 km^2) and is located 3 km below the seabed at a water depth of 65 m. The Iranian side accounts for 10% of the world and 60% of Iran's total gas reserves (U.S. Energy Information Administration (EIA)).

Furthermore, Safaniya Oil Field, the world's major offshore oilfield, is located in the Persian Gulf. Large gas finds have also been completed with Qatar and Iran sharing a huge field across the territorial median line Figure 1. By means of this gas, Qatar has built up a remarkable liquefied natural gas (LNG) and petrochemical industry.

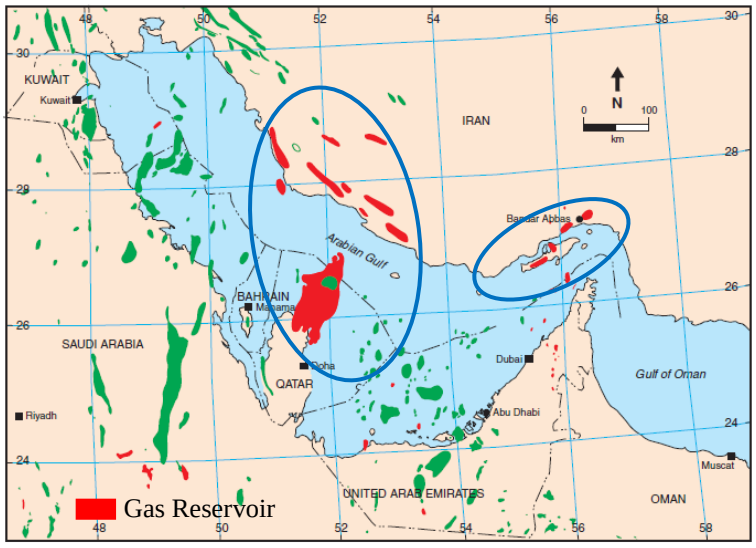

Figure 1.1 Location of gas reservoir in Persian Gulf belonged to Iran and Qatar.

1.7 Petroleum and Fingerprinting Definition

The term petroleum refers back to a family of gaseous, liquid and solid hydrocarbon materials that are both naturally occurring (crude) and man-made (refined). Crude petroleum consists of natural gas and gas condensate, light and heavy crude oil, oil shale/sand and solid bitumen; refined petroleum comprises many different fuels, solvents, petrochemicals, lubricants, waxes, asphalt and other products. Finding the source(s) of petroleum-derived contamination is often at the center of environmental

forensic investigations. The complex chemical structure of petroleum which contains tens of thousands of individual hydrocarbons and non-hydrocarbons prepare an opportunity to chemically fingerprint petroleum contamination and therewith assess its relationship to known or suspected sources. During past 15 years, considerable advances in the chemical fingerprinting of petroleum contamination for environmental forensic purposes have explored.

Although sometimes the objective of chemical fingerprinting is basically to differentiate unknown petroleum, the objective for most environmental forensic investigations including petroleum contamination is to verify the relationship between suspected source(s), impacted matrices and background samples. The discussion above has implied that chemical fingerprinting of petroleum necessarily relies upon a large number of individual diagnostic compounds or compound groups with known identities can be measured and compared.

1.8 Objectives of Study

1.8.1 General objective

To identify the Source(s) and the distribution of petroleum contaminants in soil and sediment in the vicinity of the Persian Gulf and determining major sources of PAHs in that area.

1.8.2 Specific objectives

1. To identify PAHs and *n*-alkanes contaminations and compare of different land use (Rural, Industrial, Urban, and Agricultural).

2. To characterize the source materials using molecular markers of PAHs and *n*-alkanes for various sediments (Intertidal, Offshore and River).

3. To correlate PAHs and *n*-alkane with sediment quality value.

4. To compare PAHs and *n*-alkane with sediment quality value.

5. To identify and characterize PAHs and *n*-alkane for an oil spill case occurred in Bushehr Province (Deylam port) in 2011.

6. To compare oil spill case occurred in agricultural area (Bushehr Province) with other agricultural lands.

7. To determine the distribution and correlation betweem PAHs and *n*-alkane contamination in the study area.

8. To differentiate between sources using PAHs and *n*-alkane ratios in sediment and soil samples.

1.9 Hypothesis

1. There are significant differences in PAHs and *n*-alkane between different land uses.

2. There are significant correlation between PAHs and *n*-alkane with various types of sediment.

3. There are significant differences in PAHs and *n*-alkane between different types of sediment.

4. There are significant differences in the PAHs and *n*-alkane after the oil spill with other agricultural lands.

5. There are significant correlation between PAHs and *n*-alkane ratios with various sediment and land use.

1.10 Significance of study and limitations

Southern coast of Iran is an old place to produce oil and gas, so it is necessary to control this area from the point of pollution during a long time. Since northern coast of Persian Gulf was not studied for the pollution type and quantity of each kind of these pollutants, so it is obviously important to investigate contamination level of them due to their danger for human being and organisms living there. Also, an overall research illustrating contamination level of the area was not carried out and there is not any

record for the Aliphatic, Aromatic hydrocarbon pollution. One more thing needs to be mentioned here is about PAHs and their derivatives being widespread in the environment as pollutants. They are high lipophilic chemicals so their accumulation in the body fat of living organisms is definitely probable. Moreover, they are carcinogenic or mutagenic potentially. More noticeably they are generated normally from nature and anthropogenic sources. In other word, inland spills can be much more difficult to cleanup than marine spills. It depends on a variety of factors such as local geology, soil structure, and angle of the surface, depth to ground water and access to the impacted areas. Hence, study on the polluted area on land should be more delicate and it needs detailed and comprehensive information about the area and properties of the soil and sediment. Of particular concern soil and sediment contamination from the point of petroleum spillage is a difficult job and it needs precise and accurate analysis and interpretation. As there is a vast area on the northern coasts of Persian Gulf, finding location sites and taking samples is difficult. Accordingly in each sampling day it should drive around 1000 km to collect samples so how to keep and preserve cautiously the samples is very important.

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