



TRACKING OF THE EXISTENCE OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH) IN WATER RESOURCES AROUND AND AWAY FROM AL-AHDAB OIL FIELD IN WASIT GOVERNORATE OF IRAQ

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Abstract

Some major pollutants of polycyclic aromatic hydrocarbons (PAH) those discharged as water produced (WP) from the Al-Ahdab oil field (AOF) in the ponds close to it may leak to the water resources around and eventually reaches the marshes which will affect its ecosystem. Thus, this work aims to track the availability of PAH in the water resources and the Main Outfall Drain (MOD) nearby.

The determination of PAH was evaluated using “High-Performance Liquid Chromatography (HPLC)”. The mean concentration of sixteen PAH in the produced water within the field was relatively high (0.01 to 10.89 µg/ml) with standard deviations of (0.1-0.9). While, PAH outside the field were gradually diminishes down to (0.01-0.039) x10⁻² µg/ml which exceeds the maximum contaminant level (MCL) of United States Environment Protection Agency (US-EPA) and thus leads to severe biological damages, especially since PAH reaches the (Main Drain river) is the main source of water supply for a number of wetlands like the Al-Dalmaj Marshes that is the habitat for the breeding of native Iraqi fish.

Key words : Polycyclic aromatic hydrocarbons, Al-Ahdab oil field, Al-Dalmaj Marshes, Wasit, Iraq, the Main Outfall Drain (MOD), Produced water, Wetlands, Pollution assessment.

Introduction

“Polycyclic aromatic hydrocarbons” (PAH), are commonly notorious as eco-toxicants that may harm human health (Warzecha *et al.*, 1988; B¹ kowski and Bodzek, 1998; Reese and Prest, 2002). US-EPA has been categorized as sixteen of them as precedence toxins (Kumar *et al.*, 2014; Marais *et al.*, 2016). The sixteen priority PAH includes naphthalene, dibenzo [a,h] anthracene, acenaphthylene, fluorine, chrysene, fluoranthene, Benzo[k] fluoranthene, benzo[b] fluoranthene, phenanthrene, Benz[a] anthracene, anthracene, benzo [g,h,i] perylenepyrene, benzo[a] pyrene, indeno [1,2,3-cd] pyrene, and acenaphthene accumulate in soils, sediments and in the food chain as bio-accumulate and biomagnified (Stankov Jovanovic *et al.*, 2018). Biological accumulation of PAH *in vivo* occurs in different ways like inhalation, ingestion or dermal adsorption route. US-EPA agency adopted an MCL

regulatory for the supreme carcinogen of benzo[a] pyrene not to exceed 0.2 ppb (Toxicity of Polycyclic Aromatic Hydrocarbons (PAHs): Standards and Regulations for PAHs Exposure | ATSDR - Environmental Medicine & Environmental Health Education - CSEM, 2012).

World Health Organization’s “International Agency for Research on Cancer” (IARC) has its own carcinogenic classification for each PAH compound. PAH carcinogenicity and toxicity arises when they convert metabolically to peroxides that covalently bind to DNA and other cellular molecules, and thus initiating an abnormal upsurge of DNA adducts and leading to mistakes in the replication of DNA, and consequently cancer formation in humans and other living things (Pershagen, 1990; ATSDR, 2000; Gaspari *et al.*, 2003; Rota *et al.*, 2014).

Petroleum industries and products as well as anthropogenic emissions of imperfect combustion practices, not limited to pyrogenic sources petroleum

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products and coal, are the main cause behind environmental pollution with PAH (Rota *et al.*, 2014; Abdul Kareem and Al-Janabi, 2018). Due to their low vapor pressure and high hydrophobicity, they can be found in nature in the atmosphere, mainland, and water resources.

Low molar mass PAH compounds have higher water solubility which diminishes for PAH of higher molar masses. In general, PAH has a higher tendency toward adsorption on organic substances such as humic substances, black carbon, and biopolymers. However, in natural aquatic systems, they can exist in sediments dissolved in organic materials.

Human exposure to PAH occurs mainly by the nourishment of contaminated foodstuff. The aggregate of PAH in water and sediments /soils and the close proximity to biota including humans lead to human exposure (Nam *et al.*, 2009; Tay and Biney, 2013). Therefore, the assessment and tracking of the levels of PAH presented or discharged to the environment are essential besides more attention and monitoring.

Several analytical approaches have been frequently employed for the quantification of PAH or their byproducts and derived compounds in the surroundings. Pre-dominating, chromatographic techniques like gas chromatography GC and high-performance liquid chromatography HPLC, commonly attached to mass spectrometric detection (Grosse and Letzel, 2007; Wang *et al.*, 2007; Itoh *et al.*, 2008; Onyemauwa *et al.*, 2009; Li *et al.*, 2010; Ardag *et al.*, 2011; Al-Janabi *et al.*, 2012; Kumar *et al.*, 2014). Compared to GC, HPLC systems were more appropriate for the analysis of PAH, since the low volatile compounds or the thermally unstable molecules can be easily analyzed in HPLC (Yang, 2011). Fast, reliable and precise analytical procedures are necessary for acquiescence with domestic and international regulations (Thompson *et al.*, 2002).

The aim of this work was to estimate the PAH 16 compounds from the water produced (PW) within the Ahdab oil field, a major Iraqi oil reservoir located at the southern-east of Baghdad in the province of Wasit (Alawi and Azeez, 2017), and to monitor PAH presence in the water sources (the Main Outfall Drain river) near the oil field because of its significant impact on its biodiversity in the Al-Dalmaj Marsh where it is fed as a water source.

Materials and Methods

Instruments and Reagents

- PAHs (Low Level) – WP, Certified standards (16 PAH), PE1223 SUPELCO, Sigma-Aldrich GmbH,

Germany.

- Solid-phase extraction (SPE) cartridges (Clean ENVI, 3ml×500mg, Silica Prep, Silicycle).
- HPLC system, Knauer, Berlin, Germany. The system equipped with Binary gradient system NUCLEOSIL100×5 C18 PAH, REF: 720117.40, LOT:29996032, Guard column 4mm, pressure 60 bar, Sample injector Column 20µl (autosampler), detector UV-260 nm.
- HPLC grade solvents; Methanol and acetonitrile, Sigma-Aldrich, Germany.
- Deionized water, J.T Baker, Netherland.
- Filter disks (0.45 µm), Baker bond, Darmstadt, Germany.
- PAH stock solutions were set in acetonitrile and have stored at 18°C.

Sampling

Real water samples were collected from the WP from three sites within the region of AOF in Wasit province of Iraq in addition to another five sites outside the field (Table 1). Samples were transported then preserved in the laboratory at 4°C before analysis.

Sample preparation

“Solid-phase extraction” (SPE) cartridges were used to concentrate the trace levels of PAH in water and to increase the sensitivity of the detection method. SPE was conditioned by a sequentially rinsing with 3ml of methanol, and 5ml HPLC grade water.

An aliquot of 100 ml of each sample was extracted on an SPE cartridge with a flow rate of 20 ml/min. SPE tubes were washed with 10 ml of water (HPLC grade) and kept in an oven for 30min at 70 °C to dry. The extracted PAH were retrieved from each loaded cartridge by the elution with 3 ml of acetonitrile and left aside in a fume hood to evaporate slowly and reach a final volume of 1.0 ml (Hodgeson, 1990; Cai *et al.*, 2009; Al-Janabi, 2011).

Results and Discussion

The importance of tracking the WP from AOF arises from the fact that; this oil field is large with high productivity making the monitoring process on such vast facilities and terminals hard to achieve. Nevertheless, AOF is located in the middle of agricultural land, as well as very close to the Main Outfall Drain (MOD) whose feeds several wetlands, including the Al-Dalmaj Marsh which is a land of an international reserve under Ramsar Convention because having rich biodiversity, especially with indigenous fish.

AOF discharge produced water after preliminary

Table 1: GPS coordinates for sample collection points.

#	Description of Sample		Collection Coordinates	
			Latitude(N)	Longitude(E)
1	WP within the field	Before treatment	32 49 16 1	45 66 78 6
2		After treatment	32 49 14 7	45 66 94 5
3		Evaporation ponds	32 48 79 1	45 66 78 4
4	WP outside the field (Main Drain)	Before 11 km from the field sockets	32 44 99 3	45 10 41 8
5		Before 5 km from the field sockets	32 43 63 1	45 16 05 6
6		The field sockets	32 40 33 3	45 20 66 6
7		After the field sockets	32 40 09 7	45 20 89 5
8		Hor Al-Dalj entrance	32 37 89 4	45 23 53 1

treatment. This water reaches water resources around and may reach the wetlands at the southern governorates.

This study reports results obtained from a water survey produced from the AOF in addition to an estimation of PAH levels in effluents.

Chromatographic analysis

Samples were cleaned up before injection to the HPLC appliance using a 0.45 μm filter. The identities of the compounds were established by comparing the retention time (t_r) with those obtained from the standard mixture of PAH and from spiking standards as internal references. Chromatographic conditions were; a gradient eluent of water and acetonitrile, eluents flow rate about 1 ml/min, the column oven was set to 30 $^{\circ}\text{C}$, and a total runtime of 40 min (Fig. 1). The gradient elution condition was optimized as is shown in table 2.

Chromatographic analysis for a standard mixture of PAH exhibited linearity of quantification over the range

Table 2: Gradient elution program set to separate PAH components.

Time (min)	% Water	% Acetonitrile	Flowrate (ml/min)
0.0	50	50	1.0
5.0	50	50	1.0
20.0	0	100	1.0
28.0	0	100	1.0
32.0	50	50	1.0
40.0	50	50	1.0

(0.05-15 $\mu\text{g}/\text{ml}$) for a total run time of 40min. Nonetheless, a high resolution was achieved for HPLC separation of PAH. The retention time (min), the regression equation, and the coefficients of determination (r^2) were specified for each component as listed in table 3.

The concentrations of the polycyclic compounds in water samples collected from the WP inside the AOF (samples 1 – 3) were in general within the concentration range of (0.06-10.89 $\mu\text{g}/\text{ml}$) with a standard deviation SD

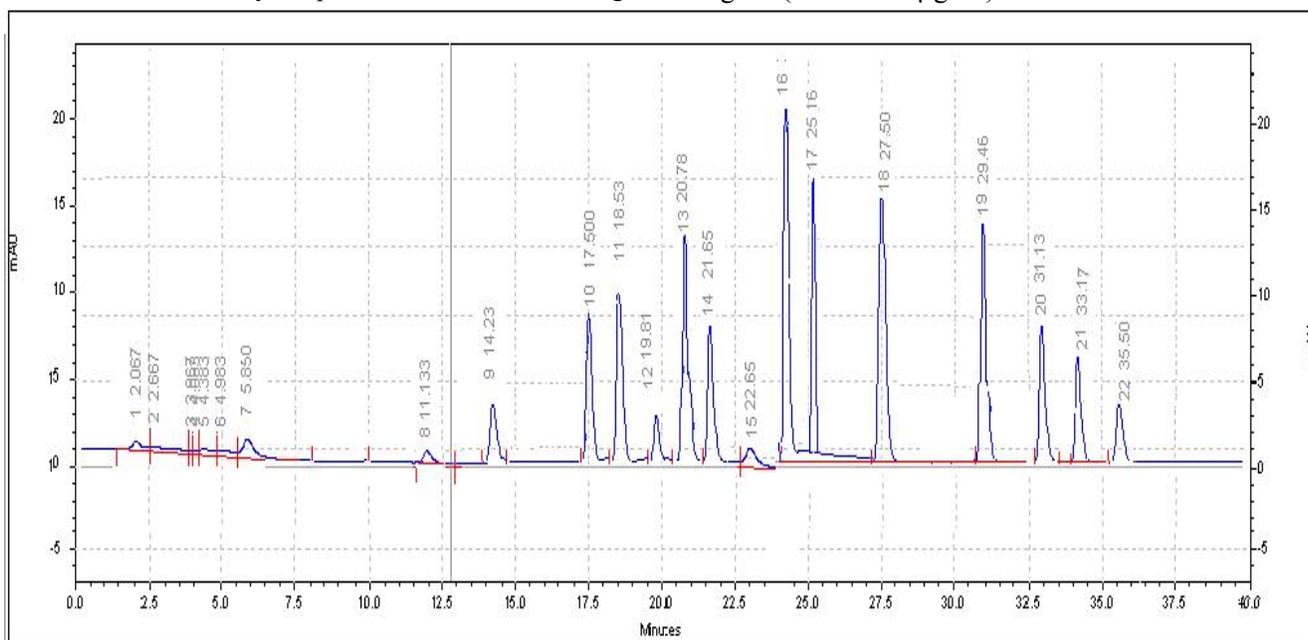
**Fig. 1:** HPLC chromatogram of a 1ppm standard PAH mixture.

Table 3: HPLC quantification of priority sixteen PAH.

PAH Compounds	Retention time (min)	Quantification ($\mu\text{g/ml}$)	Regression Equation	Linearity (R^2)
Naphthalene	5.38	0.05-15	$y = 0.987x + 0.208$	0.9994
Acenaphthylene	13.15	0.05-15	$y = 2.685x + 1.374$	0.9984
Acenaphthene	14.38	0.05-15	$y = 36.46x + 0.157$	0.9999
Fluorene	15.92	0.05-15	$y = 1.157x + 0.996$	0.9980
Phenanthrene	16.63	0.05-15	$y = 1.486x + 0.323$	0.9989
Anthracene	18.88	0.05-15	$y = 10.31x + 1.921$	0.9979
Fluoranthene	19.88	0.05-15	$y = 8.050x + 5.709$	0.9978
Pyrene	20.77	0.05-15	$y = 1.582x + 0.905$	0.9967
Benzo(a) anthracene	23.35	0.05-15	$y = 0.875x + 0.172$	0.9960
Chrysene	24.17	0.05-15	$y = 33.50x + 0.469$	0.9994
Benzo(b) fluoranthene	26.27	0.05-15	$y = 33.43x - 0.589$	0.9991
Benzo(k) fluoranthene	27.72	0.05-15	$y = 6.961x + 0.509$	0.9968
Benzo(a) pyrene	29.17	0.05-15	$y = 7.099x + 0.255$	0.9990
Dibenzo(a,h) anthracene	31.33	0.05-15	$y = 1.576x + 0.852$	0.9967
Benzo(g,h,i) pyrene	32.83	0.05-15	$y = 1.526x + 0.708$	0.9975
Indeno(1,2,3-cd)pyrene	35.07	0.05-15	$y = 1.407x + 0.324$	0.9987

Table 4: Concentration of PAH in WP from inside the AOF.

PAH Compound	Concentration $\mu\text{g/ml}$					
	Sample 1		Sample 2		Sample 3	
	Range	SD	Range	SD	Range	SD
Naphthalene	8.65-10.54	0.66	4.55- 6.61	0.9	8.75-10.89	0.9
Acenaphthylene	0.63-1.55	0.7	0.35-1.09	0.3	0.55- 1.52	0.4
Acenaphthene	0.91-1.42	0.2	0.66-1.08	0.2	1.05- 1.41	0.2
Fluorene	1.65- 3.22	0.7	0.79-2.82	0.9	1.22- 2.28	0.5
Phenanthrene	2.19-2.66	0.2	1.46-1.89	0.2	1.42- 2.31	0.4
Anthracene	0.88-1.97	0.5	0.18-1.05	0.4	0.65- 1.13	0.2
Fluoranthene	0.57-0.88	0.1	0.33-0.56	0.1	0.61-0.92	0.1
Pyrene	0.28-0.92	0.3	0.03- 0.46	0.2	0.06 - 0.87	0.3
Benzo(a) anthracene	0.06-0.82	0.4	0.02- 0.89	0.4	0.23 -1.74	0.7
Chrysene	2.84-4.40	0.8	1.35-2.23	0.4	3.97- 6.55	0.9
Benzo(b) fluoranthene	0.11-1.22	0.5	0.02-0.55	0.2	0.14 - 1.52	0.6
Benzo(k) fluoranthene	0.78-0.98	0.1	0.52-0.78	0.1	0.96-1.53	0.3
Benzo(a) pyrene	0.61-1.54	0.4	0.31-0.88	0.3	0.34-1.24	0.4
Dibenzo(a,h)anthracene	0.68-1.58	0.4	0.27-0.82	0.3	0.36-0.95	0.3
Benzo(g,h,i) pyrene	0.52-1.07	0.3	0.01-1.45	0.6	0.23-1.47	0.6
Indeno(1,2,3-cd)pyrene	0.45-0.93	0.2	0.25-0.62	0.2	0.38-0.98	0.2

ranges of (0.1-0.9) as shown in table 4.

While the concentrations of the polycyclic compounds coming from water samples collected from the WP outside the AOF (samples 5 – 8) were in the range of (0.00-0.15 $\mu\text{g/ml}$) with an SD (0.00-0.08) as it is shown in table 5.

Conclusion

The Main Outfall Drain (MOD) passes across the city of Wasit, with a very low concentration of PAH contamination, compared to PAH concentrations found in the AOF site and beyond it, *i.e.* the sockets from which the well water injection is withdrawn.

The measurements demonstrated that the concentration of most of PAH in the PW of AOF samples inside and outside exceed the standard limits allowed by the common international regulations and thus may affect the public health and the environment.

The results obtained have revealed that higher concentrations of PAH were recorded in summer, whereas the lower in winter and the contamination level with Naphthalene, Phenanthrene and Anthracene has always been dominant in most of the water samples.

Although the MOD water is not used for drinking purposes, there is a great risk of pollution, especially in the sites No.6, 7, and 8. Thousands of buffalo-producing

dairy products may cause dairy contamination and the river passes through agricultural areas, so there is a risk of these contaminants accumulate in food products to pass at the end into humans. In addition to the massive impact on the biological diversity in the wetlands and marshes.

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Table 5: Concentration of PAH in WP collected outside AOF ($\mu\text{g/ml}$).

PAH Compound	Concentration ($\mu\text{g/ml}$) $\times 10^{-2}$							
	Sample 5		Sample 6		Sample 7		Sample 8	
	Range	SD	Range	SD	Range	SD	Range	SD
Naphthalene	0.061 - 0.039	0.08	0.012-0.041	0.02	0.018 - 0.037	0.01	0.020 - 0.044	0.02
Acenaphthylene	ND - 0.074	0.03	ND - 0.076	0.03	ND - 0.078	0.02	ND - 0.088	0.03
Acenaphthene	ND - 0.064	0.03	ND - 0.066	0.02	ND - 0.069	0.02	ND - 0.073	0.02
Fluorene	0.010 - 0.079	0.03	0.015- 0.080	0.02	0.025 - 0.083	0.03	0.028- 0.085	0.02
Phenanthrene	ND - 0.061	0.02	ND - 0.071	0.02	ND - 0.083	0.06	ND - 0.087	0.02
Anthracene	0.032 - 0.086	0.03	0.036 - 0.085	0.02	0.038 - 0.088	0.02	0.039- 0.092	0.02
Fluoranthene	ND - 0.058	0.03	ND - 0.059	0.02	ND - 0.067	0.03	ND - 0.068	0.03
Pyrene	ND - 0.087	0.03	ND - 0.088	0.03	ND - 0.093	0.03	ND - 0.098	0.04
Benzo(a) anthracene	ND -0.042	0.01	ND - 0.043	0.01	ND - 0.055	0.02	ND - 0.067	0.03
Chrysene	0.048- 0.131	0.03	0.052- 0.135	0.03	0.059 - 0.145	0.04	0.061- 0.150	0.04
Benzo(b) fluoranthene	ND -0.071	0.04	ND - 0.075	0.04	ND - 0.078	0.04	ND -0.082	0.03
Benzo(k) fluoranthene	ND -0.067	0.04	ND - 0.072	0.06	ND - 0.076	0.05	ND -0.081	0.05
Benzo(a) pyrene	ND -0.032	0.02	ND -0.038	0.02	ND - 0.043	0.02	ND - 0.052	0.03
Dibenzo(a,h) Anthracene	ND -0.033	0.07	ND - 0.040	0.01	ND - 0.044	0.01	ND - 0.051	0.05
Benzo(g,h,i) pyrene	ND -0.034	0.02	ND - 0.045	0.02	ND - 0.045	0.02	ND - 0.051	0.02
Indeno(1,2,3-cd) pyrene	ND	–	ND	–	ND	–	ND	–

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