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ORIGINAL PAPER

Levels, spatial distributions, and provision of petroleum hydrocarbons and phthalates in sediments from Obhur lagoon, Red Sea coast of Saudi Arabia

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Abstract The levels, spatial distribution, and sources of petroleum hydrocarbons and phthalates were assessed in surface sediment samples from the urban lagoon of Obhur near Jeddah, the largest city on the Red Sea coast of Saudi Arabia. The lagoon was divided into the inner zone, middle zone, and outer zone based on its geomorphological features and developmental activities. n-Alkanes, hopane and sterane biomarkers, and unresolved complex mixture were the major petroleum hydrocarbon compounds of the total extractable organic matter. Phthalates were also measured in the sediment samples. In the three zones, n-alkanes ranged from 89.3 ± 88.5 to 103.2 ± 114.9 ng/g, whereas the hopane and sterane biomarkers varied from 69.4 ± 75.3 to 77.7 ± 69.9 ng/g and 72.5 ± 77.9–89.5 ± 82.2 ng/g, respectively. The UCM concentrations ranged from 821 ± 1119 to 1297 ± 1684 ng/g and phthalates from 37.4 ± 34.5 65 ± 68 ng/g. The primary origins of these anthropogenic hydrocarbons in the lagoon sediments were petroleum products (boat engine discharges, boat washing, lubricants, and wastewater flows) and plasticizers (plastic waste and litter). The proportions of anthropogenic hydrocarbons derived from petroleum products in the sediment's TEOM ranged from 43 ± 33 to 62 ± 15%, while the percentages for plasticizers varied from 2.9 ± 1.2 to 4.0 ± 1.6%. The presence and inputs of these contaminants from petroleum and plastic wastes in the lagoon's sediments will eventually have an impact on its habitats, including the benthic nursery and spawning areas.

Keywords Obhur lagoon · Saudi Arabia · Red Sea · Hydrocarbons · Phthalates · UCM · GC–MS

Introduction

Coastal areas are mostly affected by anthropogenic sources from petroleum hydrocarbons (Gao et al., 2022; Ndungu et al., 2017; Uddin et al., 2021). These sources include onshore oilfield operations, liquid released by refineries, accidental spills, petrochemical plants and shipping, natural oil seeps, and sewage

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discharges (Alsharhan, 2003; El Nemr et al., 2004; El Sayed, 2002a, 2002b; Rushdi et al., 2009, 2022a). Petroleum residues in the Red Sea are largely attributed to oil tankers and oil terminals (Dicks, 1987; El-Sikaily et al., 2003; Rushdi et al., 2010), where the coastal ecosystems of the Red Sea shorelines are seriously contaminated with oil-related products (Alhudhodi et al., 2022; Jamoussi et al., 2022; Kostianaia et al., 2020; Periáñez, 2020; Rushdi et al., 1994; Shetaia et al., 2016). In the coastal environment of Saudi Arabia's Red Sea, oil pollution can also originate from sewage, leisure activities, and littering (El Sayed, 2002a, 2002b; Wilson et al., 2017). Sewage is frequently discharged to the coastal zones, especially into nearby lagoons, around large cities such as the city of Jeddah (Badr et al., 2009). It is also discharged from offshore platforms, ship traffic, tour boats, ferries, and private yachts (Hees, 1977). Transported atmospheric particulate matter, wind-resuspended soil, and seasonal runoff and floods can significantly contribute to petroleum-related products in the coastal environment (Rushdi et al., 2010, 2022b). The marine life of the distinct ecosystems of the Red Sea coast is facing significant danger due to contamination from oil-related sources (Batang et al., 2016; UNEP, 1985; Ziegler et al., 2016).

Jeddah has a population of about 4.7 million, making it the biggest city on the western coast of Saudi Arabia. It is projected to reach 5.3 million in 2023 (WPR, 2023). The city is highly industrialized and hosts one of the largest Saudi Aramco refineries. However, the presence of other facilities, including municipal waste and sewage, electrical power generation, refinery plants, and desalination, has negative effects on the environment along the shoreline (Badr et al., 2009). According to (Durmus, 2019), approximately 40% of the total 1.9 million tons of solid waste in the city is organic waste. Unfortunately, some of this waste can end up polluting the environment and reaching the coastlines through runoff or particulate matter transported by wind (Al-Khion, et al., 2021; Al-Lihaibi, et al., 2019; Rushdi et al., 2010, 2022a, 2022b). Plastics, which make up most of the marine litter, are a major source of contaminants in the marine environment (Diem et al., 2023; Ryan, 2015) and have different impacts on marine biota based on the size of the plastic litter (Barboza et al., 2019; Bellou et al., 2021). Obhur Creek near Jeddah, also known as Sharm Obhur, is a well-known tourist spot situated on the western coast of Saudi Arabia. It acts as a link between inland and marine ecosystems and provides visitors with resorts, harbors, docks, and recreational activities like boat trips. Nevertheless, the water outflow from the lagoon can introduce land source pollutants to the Red Sea.

The concentrations and contribution of anthropogenic petroleum hydrocarbons and plastic-related compounds in the sediment of the lagoon are still unknown. Therefore, the main objectives of this work are to: (1) determine the levels and spatial distribution of n-alkanes, hopanes, steranes, and phthalates in sediments from Sharm Obhur on the Red Sea coast of Saudi Arabia; (2) identify their potential sources in the sediments based on biomarkers and geochemical indices; and (3) describe their possible environmental impacts.

Experimental

Study area, sampling sites, and sample preparation

The study area (Fig. 1) is Obhur Lagoon (also locally known as Sharm Obhur). It is located about 35 km north of Jeddah City and is around 11 km long and 1.5 km wide, with a depth of 35 m near its mouth on the Red Sea and about 6 m near the head (Rasul, 2015). Sharm Obhur is a highly favored recreational spot in Jeddah, known for its abundance and popularity. It is connected to the Red Sea proper through a narrow outlet, spanning 264 m in width, situated at its southwestern end. According to Basaham et al., (2006), the hydrographic structure displays a two-layer flow pattern at its entrance. The incoming water is low in salinity and occurs at both surface and intermediate depths. The outgoing water is more saline and found at the bottom. The water temperature and salinity range from 24.4 °C to 32.2 °C and from 39.10 to 40.2 between winter and summer, respectively (Alsaafani et al., 2017; Basaham & El-Shater, 1994). The inflow of surface water from the sea to the lagoon is about 30 psu salinity, whereas the outflow of deep water to the sea from the lagoon is about 39 psu (Abdulla & Al-Subhi, 2020; Albarakati, 2009; Alsaafani et al., 2017). The amount of water coming into the area varies between approximately 151 m^3/sec in November and 302 m^3/sec in February. Similarly, the amount of water flowing out of the area

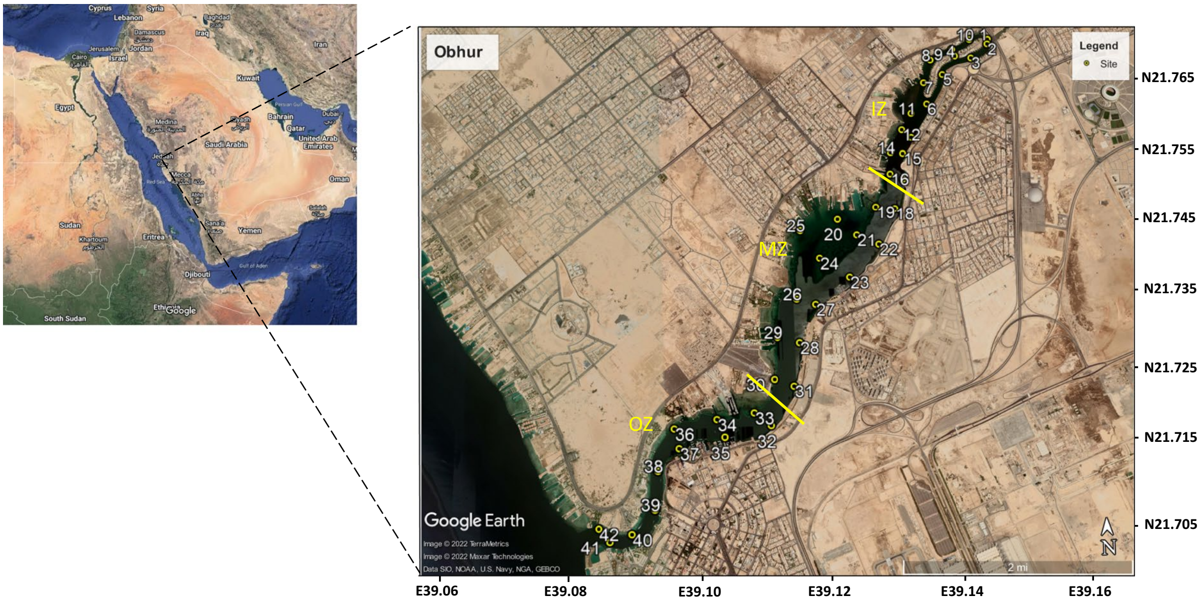

Fig. 1 Location map showing the sampling sites of the inner zone (IZ), middle zone (MZ), and outer zone (OZ) in the Obhur lagoon, Saudi Arabia

ranges from about 151 m^3/sec in November to 402 m^3/sec in February (El-Rayis & Eid, 1997).

Surface sediment samples from forty-two stations were collected from the lagoon in May 2014 (Fig. 1) by Van Veen grab samplers where thirty stations covering and representing the different zones and the entire lagoon were selected and chemically analyzed. The samples were stored in ice boxes and transferred to a freezer in the lab after 4–5 h. About 10 g from each sediment sample was taken by a clean spatula, defrosted, and dried at room temperature, then ground and sieved to obtain < 125 µm fine particles. We divided the lagoon into three zones based on its physical feature and developmental activities (Fig. 1). They were the inner inland zone (IZ), middle zone (MZ), and outer zone (OZ).

Extraction and Instrumental analysis

150 mL precleaned beakers were used for the sediment sample extraction. About 5 g of each sieved sediment sample was extracted with a mixture of dichloromethane (DCM = 30 ml) and methanol (MeOH = 10 ml) three times (15 min each) using ultrasonic agitation (Rushdi et al., 2014a, 2014b, 2018). A filtration unit containing an annealed glass fiber filter was used to remove the sediment particles. First, the filtrate was concentrated by a rotary evaporator and then reduced to about 200 µL by a flow of dry nitrogen gas. Then, the volume of the total extract was corrected to exactly 500 µL by adding DCM:MeOH (3:1, v:v) mixture.

Gas chromatography-mass spectrometry (GC–MS) with a Hewlett-Packard 6890 GC coupled to a 5975 Mass Selective Detector (Agilent) was used for total extract analysis. An Agilent DB-5MS fused silica capillary column (30 m long, 0.25 mm internal diameter, and 0.25 um film thickness) and helium as carrier gas were used. The setting of the GC oven temperature was 65 °C with an initial hold for 2 min, then increased to 310 °C at 6 °C/min and isothermal final hold for 20 min. The ion source energy for the MS was the electron impact mode at 70 eV. The mass spectrometric data were acquired using the ChemStation data system.

Identification, quantification, and quality control

The hydrocarbon compounds identification was based on the similarity of the retention times of each compound with the external standard and the GC–MS data. The identifications of n-alkanes, hopanes, steranes, and phthalates are based principally on GC retention times and their key ion patterns and mass spectra (i.e., key ion fragmentograms *m/z* 85, 191, 217/218, and 149, respectively). The quantification was performed from the GC profiles utilizing the external standard method with authentic compounds of each homologous series (Rushdi et al., 2006, 2010, 2014a). The external standards were n-alkanes (C_8–C_{40} (90–100%); Supelco^R -Sigma-Aldrich, USA)), hopanes (17α(H),21β(H)-hopane, $C_{30}H_{52}$; (90–100%); Supelco^R -Sigma-Aldrich, USA)), and bis(2-ethylhexyl)phthalate; (100%), Sigma-Aldrich, USA). The average response factors were computed for each compound. The peak areas of the compounds derived from the TIC traces were used for quantifications.

During the course of this study, the quality control consisted of analytical chemistry and biomarker method application. n-Tetracosane-d50 was added to both the fiber filter and sediment samples to test recoveries, which were 92.8% and 72.9%, respectively, and the measured concentrations were adjusted accordingly. We measured the limit of detection (LoD) and limit of quantification (LoQ) the same way with the samples where the least-square method was applied to fit the relative responses of the different standards versus their concentrations. The correlations were significant with correlation coefficients (R^2 = 0.91–0.98). The LODs were 0.04–0.7 ng/μL for n-alkanes, 0.1–1.5 ng/μL for Hopane, and 0.1–1.6 ng/μL for phthalate. We have tested all reagents and solvents used for extraction for possible contaminations. Procedural blanks were examined to assess background contamination introduced by laboratory practices. Throughout the entire procedure, we furthermore performed blank extracts after batches of three samples.

Results and discussion

The sediment samples had a range of total extractable organic matter (TEOM) concentrations from 119 to 5924 ng/g (Fig. 2a). The average values were 1428 ± 1448 ng/g for the inner/inland zone (IZ), 1982 ± 1710 ng/g for the middle zone (MZ), and 2080 ± 2229 ng/g the outer zone (OZ), as shown in Table 1, and Fig. 3a. The data revealed that there were significant amounts of hydrocarbon compounds, including biomarkers, present in the TEOM (Table 1.). The detected compounds included n-alkanes, hopane and sterane biomarkers, phthalates, and an unresolved complex mixture (UCM) of highly branched and cyclic hydrocarbons (Table 1 and SM1), which are the emphasis of the discussion in this work.

The existence, dispersal patterns, amounts, and characteristics of homologous series of n-alkanes, hopanes, and steranes in the environment can be applied to identify their major sources in the environment (; Bouloubassi et al., 2001; Rushdi et al., 2006, 2010, 2014a; Simoneit, 1977a, 1984, 1985). Therefore, similarities are likely between the observed organic compound mixtures in the environmental samples and the recognized sources of those compounds. Here, merely non-polar hydrocarbons and phthalates in the TEOM are explained and discussed.

n-Alkanes

The sediment samples' TEOM contained n-alkanes ranging from C_{17} to C_{34} (Table 1), with the highest concentration found at C_{31} (hentriacontane). The total concentration ranged from 5.7 ng/g to 464.6 ng/g, with average values of 140.6 ± 196.4 ng/g in sediments from IZ, 89.3 ± 88.5 ng/g in MZ, and 103.2 ± 114.9 ng/g in OZ (Table 1 and MS1, Fig. 3b). The IZ had the highest concentration range followed by OZ and MZ as shown in Fig. 2a, indicating that the topography and hydrography of the lagoon, as well as human activities around it, impact the distribution of these alkanes. These concentrations were relatively similar to the levels measured in Jiaozhou Bay of China (0.5–8.2 μg/g; Wang et al., 2006), Gulf of Suez-Egypt (34–553 ng/g; El Nemr et al., 2014), Yellow River estuary-China (0.356–0.572 mg/kg; Wang et al., 2018), Patos Lagoon estuary-Brazil (0.28–36.4 μg/g; Neves et al., 2023). But they were lower than the values reported in other regions such as the Caspian coast-Iran (249–3900 μg/g; Shirneshan et al., 2016), Eastern Mediterranean Coast (1.6 14.7 μg/g; Tsapakis et al., 2010), Northern Arabian Gulf (5654–20,942 ng/g; Rushdi et al., 2014b).

The sources of n-alkanes in the environment are mainly biogenic and anthropogenic contributions, and their sources can be identified based on the distribution pattern of their homologous series. The existence and detection the n-alkanes in ecosystems are valuable markers to assess the sources, transport, and

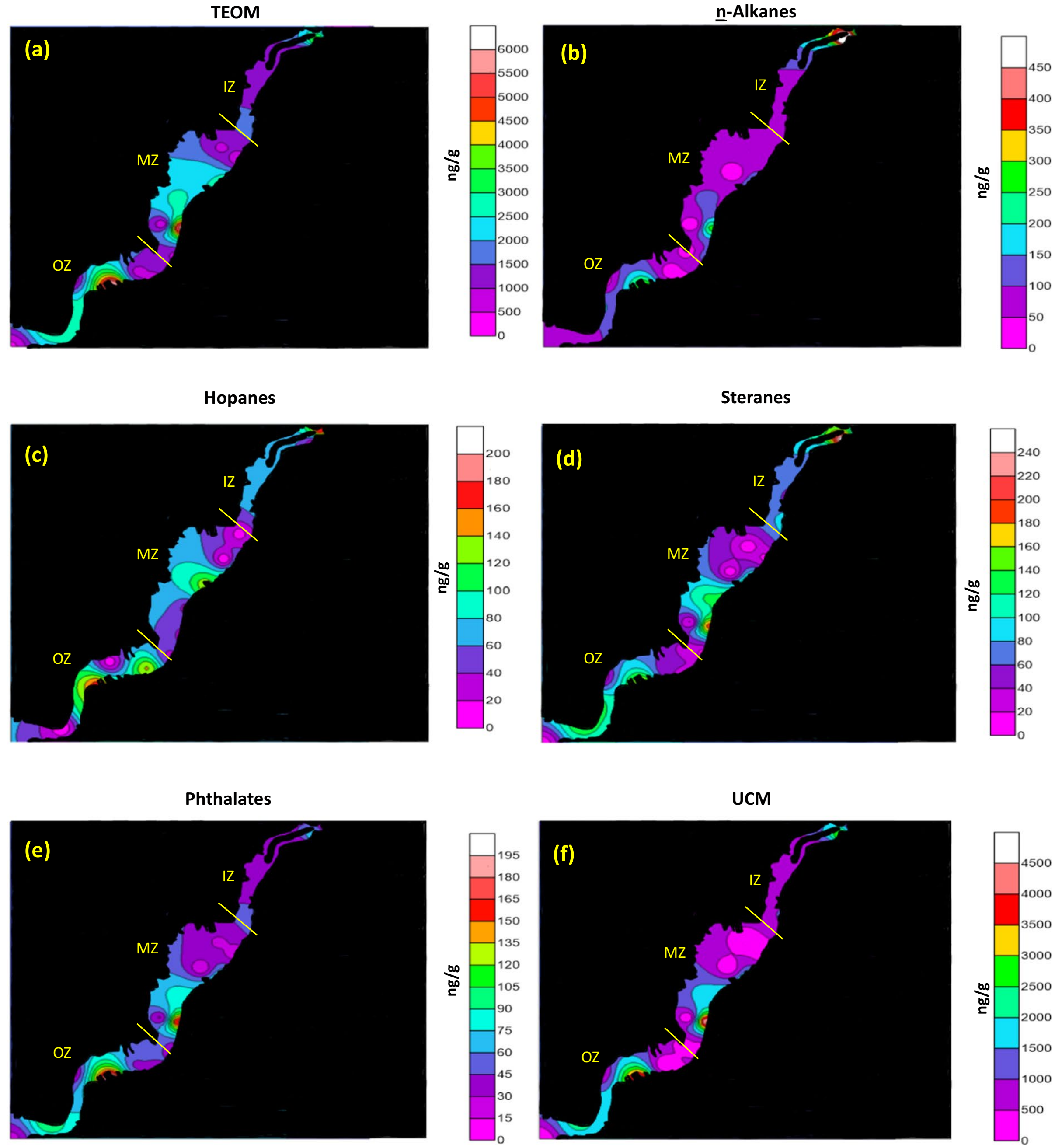

Fig. 2 Spatial concentration distributions of **a** TEOM, **b** n-alkanes, **c** hopanes, **d** steranes, **e** phthalates, and **f** UCM in sediments from the Obhur lagoon

preservation of organic matter in the environment. The focal parameters coupled with the n-alkane characteristics and sources are the carbon number maximum (C_{max}) of the most abundant n-alkane in the homologous series and the carbon preference index (CPI; Bray & Evans, 1961). The $CPI_{o/e}$ (Mazurek & Simoneit, 1984) was estimated using the equation:

$$CPI_{(o/e)} = \text{Total nC}_{odd}/\text{Total nC}_{even}$$

Table 1 The average concentrations (Avg.) and standard deviation (SD) in ng/g of TEOM, n-alkanes, hopanes, steranes, and UCM in sediments samples from the three zones (IZ, MZ, and OZ) of Obhur lagoon of Saudi Arabia and their related organic geochemical parameters

			IZ		MZ		OZ	
			Avg	SD	Avg	SD	Avg	SD
TEOM (ng/g)			1428	1448	1982	1710	2080	2229
Compound								
n-Alkanes (ng/g)	**Composition**	**M.W**						
Heptadecane	$C_{17}H_{36}$	240	2.5	5.3	0.9	1.1	1.7	2.2
Octadecane	$C_{18}H_{38}$	256	2.5	4.4	1.8	1.6	2.8	2.6
Nonadecane	$C_{19}H_{40}$	268	5.6	10.8	3.4	4.0	5.9	4.7
Eicosane	$C_{20}H_{42}$	282	7.1	12.2	3.6	4.9	2.6	2.9
Heneicosane	$C_{21}H_{44}$	296	8.2	15.2	5.6	5.2	10.3	13.4
Docosane	$C_{22}H_{46}$	310	11.1	19.1	3.9	3.8	4.7	4.6
Tricosane	$C_{23}H_{48}$	324	7.9	12.0	5.5	4.8	8.3	11.3
Tetracosane	$C_{24}H_{50}$	338	7.3	11.2	4.4	4.7	7.9	10.5
Pentacosane	$C_{25}H_{52}$	352	7.6	9.8	5.8	5.0	7.9	9.0
Hexacosane	$C_{26}H_{54}$	366	8.6	14.1	6.8	7.8	3.8	3.5
Heptacosane	$C_{27}H_{56}$	380	9.7	10.5	7.2	7.5	8.6	11.5
Octacosane	$C_{28}H_{58}$	394	7.6	14.7	3.8	4.7	3.8	4.0
Nonacosane	$C_{29}H_{60}$	408	11.6	12.1	7.4	9.2	9.8	13.0
Triacontane	$C_{30}H_{62}$	422	7.3	14.1	4.0	5.1	3.7	4.3
Hentriacontane	$C_{31}H_{64}$	436	24.6	21.1	18.3	22.6	17.2	24.8
Dotriacontane	$C_{32}H_{66}$	450	5.1	12.5	2.6	3.4	2.0	2.6
Tritriacontane	$C_{33}H_{68}$	464	4.5	5.6	3.0	3.6	1.6	2.3
Tetratriacontane	$C_{34}H_{70}$	478	2.0	5.5	1.4	1.3	0.6	0.7
Total			**140.6**	**196.4**	**89.3**	**88.5**	**103.2**	**114.9**
CPI (o/e)[a]			2.0	0.7	1.9	0.9	2.3	1.7
Wax n-Alkanes (ng/g)								
Pentacosane	$C_{25}H_{52}$	352	0.7	1.2	0.9	1.0	2.3	3.2
Heptacosane	$C_{27}H_{56}$	380	3.6	4.8	2.2	3.4	5.1	9.3
Nonacosane	$C_{29}H_{60}$	408	5.8	7.2	3.7	4.5	6.0	9.0
Hentriacontane	$C_{31}H_{64}$	436	18.4	18.9	15.0	19.9	14.3	22.4
Tritriacontane	$C_{33}H_{68}$	464	2.4	4.2	1.6	2.5	0.7	1.8
Total			31.2	34.4	23.4	29.0	28.5	45.1
Total Petroleum n-alkanes (ng/g)			**109.4**	**191.9**	**65.9**	**65.8**	**74.7**	**72.0**
Total Petroleum n-alkanes (%)			**62.7**	**15.3**	**75.0**	**17.3**	**76.7**	**21.2**
Hopane Biomarkers (ng/g)								
Trisnorneohopane	$C_{27}H_{46}$	370	4.0	3.2	4.1	4.3	4.7	4.5
17α(H)-Trisnorhopane	$C_{27}H_{46}$	370	8.3	7.5	11.7	13.9	11.8	12.1
17α(H), 21β(H)-Norhopane	$C_{29}H_{50}$	398	13.7	11.2	12.5	13.4	14.1	13.2
Trisnorhopane	$C_{29}H_{50}$	398	3.9	2.8	3.4	3.8	3.7	3.4
17 α (H), 21β(H)-Hopane	$C_{30}H_{52}$	412	13.5	10.4	12.0	12.6	13.6	12.0
17α(H), 21β(H)-22S-Homohopane	$C_{31}H_{54}$	426	7.4	5.4	6.8	7.4	7.0	5.7
17α(H), 21β(H)-22R-Homohopane	$C_{31}H_{54}$	426	4.9	3.0	3.9	4.2	4.4	3.6
Gammacerane	$C_{30}H_{52}$	412	0.6	0.9	0.2	0.4	0.3	0.2
17α(H), 21β(H)-22S-Bishomohopane	$C_{32}H_{56}$	440	4.8	3.5	4.0	4.4	4.9	4.6
17α(H), 21β(H)-22R-Bishomohopane	$C_{32}H_{56}$	440	3.9	3.3	3.5	3.8	4.1	3.2

Table 1 (continued)

			IZ		MZ		OZ	
			Avg	SD	Avg	SD	Avg	SD
17α(H), 21β(H)-22S-Trishomohopane	$C_{33}H_{58}$	454	2.9	2.1	2.3	2.4	2.6	2.2
17α(H), 21β(H)-22R-Trishomohopane	$C_{33}H_{58}$	454	1.6	1.3	1.3	1.4	1.8	1.6
17α(H), 21β(H)-22S-Tetrakishomohopane	$C_{34}H_{60}$	468	1.7	1.0	1.2	1.2	1.3	1.3
17α(H), 21β(H)-22R-Tetrakishomohopane	$C_{34}H_{60}$	468	1.3	0.9	0.8	0.9	1.0	1.1
17α(H), 21β(H)-22S-Pentakishomohopane	$C_{35}H_{62}$	482	1.7	1.1	1.1	1.5	1.5	1.4
17α(H), 21β(H)-22R-Pentakishomohopane	$C_{35}H_{62}$	482	0.7	0.4	0.5	0.6	0.8	0.8
Total			**74.9**	**54.6**	**69.4**	**75.3**	**77.7**	**69.9**
C_{31} S/(R + S)			0.56	0.11	0.65	0.06	0.62	0.05
C_{32} S/(R + S)			0.57	0.15	0.53	0.07	0.53	0.07
Steranes (ng/g)								
13β, 17α-20S-Cholestane Diasterane	$C_{27}H_{48}$	372	5.9	5.8	4.9	4.8	6.9	6.6
13β, 17α-20R-Cholestane Diasterane	$C_{27}H_{48}$	372	3.5	3.4	2.9	2.8	3.8	3.2
13β, 17β-20S-Cholestane Diasterane	$C_{27}H_{48}$	372	3.1	3.0	2.3	2.5	3.3	3.7
13β, 17β-20R-Cholestane Diasterane	$C_{27}H_{48}$	372	4.0	4.7	4.1	5.5	5.9	6.6
14α, 17βα-20S-Cholestane	$C_{27}H_{48}$	372	5.0	4.8	3.9	4.5	4.6	4.4
14β,17β -20R-Cholestane	$C_{27}H_{48}$	372	6.7	6.3	5.1	5.5	5.8	5.7
14β, 17β-20S-Cholestane	$C_{27}H_{48}$	372	10.4	9.8	7.7	8.1	9.4	9.4
14α,17βα-20R-Cholestane	$C_{27}H_{48}$	372	4.6	4.4	1.8	1.7	2.0	2.6
14α, 17βα-20S-Ergostane	$C_{28}H_{50}$	386	2.6	3.0	2.2	2.9	3.8	4.0
14α, 17β-20R-Ergostane	$C_{28}H_{50}$	386	7.7	7.4	6.7	7.3	7.6	7.0
14α, 17β-20S-Ergostane	$C_{28}H_{50}$	386	3.2	3.0	2.5	2.6	3.3	3.6
14α, 17βα-20R-Ergostane	$C_{28}H_{50}$	386	0.8	0.6	0.7	0.8	0.4	0.3
14α, 17βα-20S-Stigmastane	$C_{29}H_{52}$	400	8.8	8.9	7.4	9.3	8.0	8.0
14β, 17β-20R-Stigmastane	$C_{29}H_{52}$	400	12.0	11.4	10.0	11.4	8.4	7.0
14β, 17β-20S-Stigmastane	$C_{29}H_{52}$	400	7.3	6.5	7.0	7.4	6.2	6.4
14α, 17βα-20R-Stigmastane	$C_{29}H_{52}$	400	3.9	3.4	3.3	4.0	2.2	2.0
Total			**89.5**	**82.2**	**72.5**	**77.9**	**81.4**	**76.8**
C27 S/(R + S)			0.48	0.11	0.42	0.12	0.46	0.10
C29 S/(R + S)			0.65	0.15	0.62	0.28	0.66	0.22
Phthalates (ng/g)								
Di-isobutyl phthalate	$C_{16}H_{22}O_4$	278	5.5	7.2	14.5	11.9	17.1	19.6
Dibutyl phthalate	$C_{16}H_{22}O_4$	278	4.8	5.7	10.0	9.1	11.2	11.1
Dioctyl phthalate	$C_{24}H_{38}O_4$	390	27.0	23.0	34.2	29.0	36.7	37.6
Total			**37.4**	**34.5**	**58.6**	**49.0**	**65.0**	**68.0**
UCM			**821**	**1119**	**1041**	**1539**	**1297**	**1684**
U:R			1.00	0.99	0.86	1.17	1.23	1.33

Bold indicates total concentrations of each group

The major samples had maximum concentrations at C_{31} (Table 1 and SM1), suggesting a contribution from higher terrestrial plant waxes of grassy flora (Eglinton & Hamilton, 1967; Ficken et al., 2000; Simoneit, 1978; Zhang et al., 2006). This higher molecular weight n-alkane was also detected in the sediments from the Arabian Gulf (Rushdi et al., 2010, 2022a) signifying that plant waxes of tropical vegetation have a high C_{max} (Simoneit, 1978). Frequently, the n-alkane $CPI_{(o/e)}$ has been employed to assess the impact and influence of biogenic and anthropogenic inputs (Al-Khion et al., 2021; Bray & Evans, 1961;

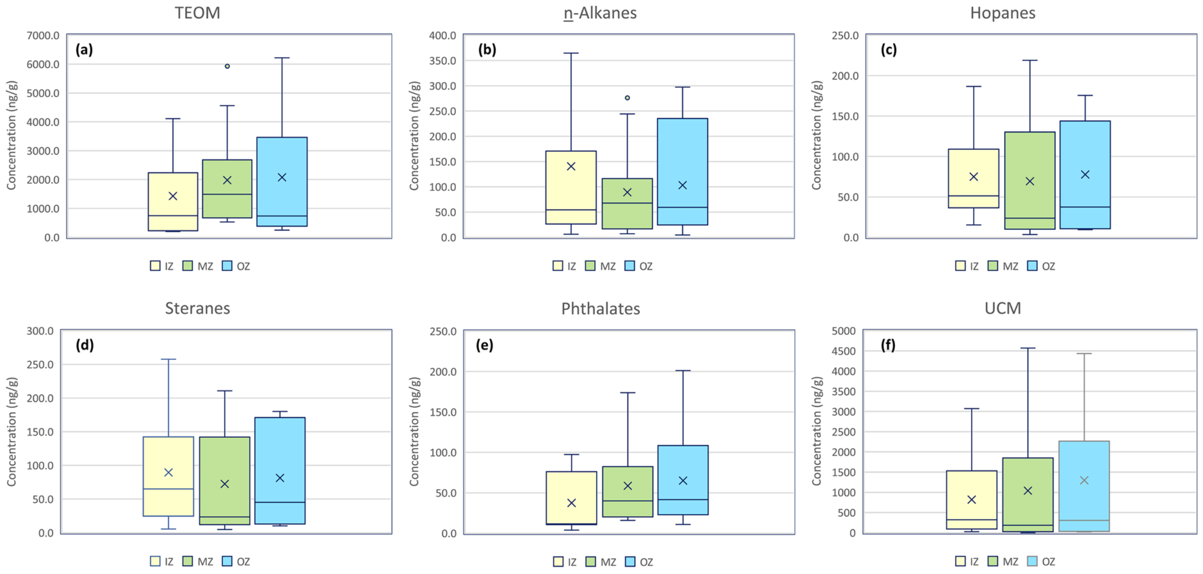

Fig. 3 Box plots of the concentrations of **a** TEOM, **b** n-alkanes, **c** hopanes, d steranes, **e** phthalates, and **f** UCM in sediments from the inner zone (IZ), middle zone (MZ), and outer zone (OZ) of Obhur lagoon

Colombo et al., 1989; Commendatore & Esteves, 2004; Diefendorf et al., 2014; Rushdi et al., 2022a; Simoneit, 1989). In the sediment of the lagoon, the $CPI_{(o/e)}$ values for n-alkanes with carbon chains ranging from C_{17}–C_{35} were estimated between 0.8 and 5.7 (Table SM1). Specifically, the values for sediment in IZ ranged from 0.8 to 3.5 (average = 2.0 ± 0.7), for MZ it was 1.1 to 4.4 (average = 1.9 ± 0.9), and for OZ it was 1.1 to 5.7 (average = 2.1 ± 1.7) as shown in Table 1. These values confirmed that n-alkanes in the sediments were originally derived predominantly from petroleum-related products with small amounts from higher plant waxes.

To assess the relative inputs of the different sources, the n-alkane concentrations from plant wax were calculated following the method of Simoneit et al., (1991a). The plant wax concentrations varied from 1.1 to 132 ng/g of total n-alkanes of the lagoon. They were ranging from 3.1 to 98.5 ng/g (average = 31.2 ± 34.4 ng/g) in the IZ, 0.6–60.1 ng/g (average = 23.4 ± 29.0 ng/g) in the MZ, and from 1.5 to 57.2 ng/g (average = 28.5 ± 45.1 ng/g) in the OZ (Table 1 and SM1). The TAR_{alk} ratios (terrestrial-to-marine n-alkanes; $TAR_{alk} = [(nC_{27} + nC_{29} + nC_{31})/(nC_{15} + nC_{17} + nC_{19})]$; Bourbonniere & Meyers, 1996) were calculated and found to range from 9.3 ± 10.1 for ZII to 28.2 ± 22.4 for ZI (Table SM1). These high values indicated that terrestrial plant sources were prevalent in these sediments. The petroleum n-alkane inputs were then estimated by deducting the calculated plant wax n-alkanes from the total n-alkane concentrations. Their total concentrations ranged from 4.0 to 444.2 ng/g in the sediment samples of the lagoon. They ranged from 4.0 to 444.2 ng/g (average = 109.4 ± 191.9 ng/g) in the IZ, 7.8–188.6 ng/g (average = 65.9 ± 65.8 ng/g) in MZ, and from 2.7 to 272.7 ng/g (average = 74.4 ± 72.0 ng/g) in the OZ (Table 1 and SM1). The relatively low concentrations of n-alkanes and the absence of the isoprenoid pristane and phytane in the lagoon sediments are probable due to biodegradation and high oxidation processes.

Hopanes and steranes

Both hopane and sterane biomarkers were detected in the lagoon sediment samples. Hopanes, which were observed to range from C_{27} to C_{35} had C_{max} at 29 and 30 (Table 1), were measured with concentrations ranging from 4.2 to 197.0 ng/g (Table SM1). As shown in Table 1 and Figs. 2c, 3, the average concentrations were roughly similar in the three zones (i.e., 74.9 ± 54.6 ng/g in IZ, 69.4 ± 75.3 ng/g in MZ, and 77.7 ± 69.9 ng/g in OZ). These concentrations were in the range of the levels reported in the sediments of the estuary of Paranagaue Bary-Southeast Atlantic (41.2–198 ng/g; Garcia et al., 2019); they were higher than the concentrations in the coastal sediments of Qatar (0.0–32.9 ng/g; Rushdi et al., 2017a, 2017b), and lower than the values determined in the coastal canal of Thailand (1510–17114 ng/g; Boonyatumanond et al., 2006), Iranian coast of Arabian/Persian Gulf (189–3713 ng/g, 42–3864 ng/g; Jafarabadi et al., 2019), and estuary in Malaysia (023–2.45 mg/kg; Keshavarzifard et al., 2022) Steranes total concentrations ranged from 6.2 to 194.3 ng/g with averages of 89.5 ± 82.2 ng/g, 72.5 ± 77.9 ng/g, and 81.4 ± 76.8 ng/g for sediments from IZ, MZ, and OZ, respectively (Figs. 2d and 3d). They ranged from C_{27} to C_{29} with a C_{max} at 29 and major amounts at C_{27} (Table 1). The concentrations of these sediments were lower than the levels measured in the sediments of the Arabian/Persian coast of Iran coast (42–3864 ng/g; Jafarabadi et al., 2019), similar to the values in Leizhou Peninsula-South China (20–230 ng/g; Gao et al., 2021), and relatively higher than the levels in the coastal zone of Qatar (0.7–36.3 ng/g; Rushdi et al., 2017a, 2017b).

The occurrence of hopane and sterane biomarkers in the environment suggests that the source of organic matter was fossil fuel residues (e.g., Jafarabadi et al., 2019; Medeiros et al., 2005; Simoneit, 1984; Simoneit et al., 1991b). The positive correlations between hopane and sterane concentrations versus TEOM were significant (Fig. 4; $R^2 = 0.74–0.85$, 0.69–0.72, and 0.70–0.8 for IZ, MZ, and OZ respectively),

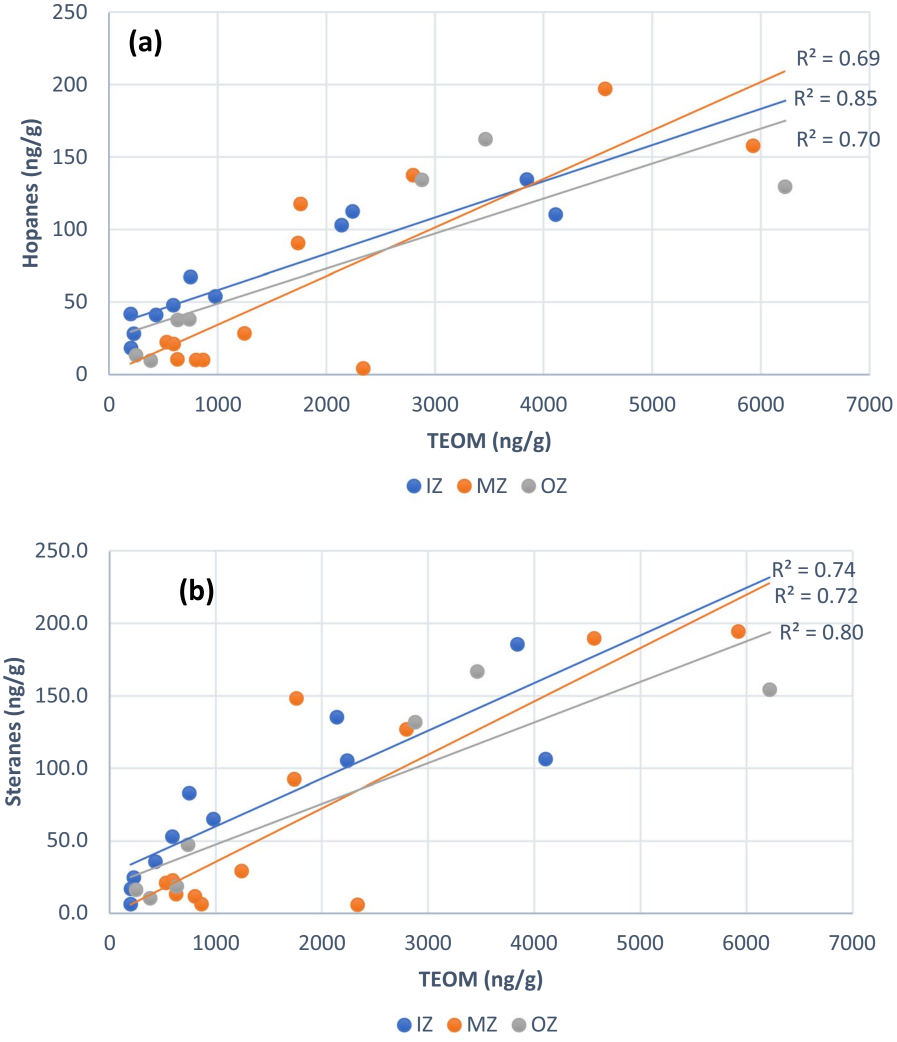

Fig. 4 Correlation plots showing the relationships between: **a** concentrations of hopanes and **b** steranes versus total concentrations of extractable hydrocarbons (TEOM) for the three zones

confirming that petroleum residues were also major sources to the sediment of the lagoon. Hopanes are usually applied as biomarker tracers for fossil fuel contributions in the environment since they resist alteration and degradation processes (Ten Bouloubassi et al., 2001; Haven et al., 1988; Moldowan et al., 1985; Peters & Moldowan, 1993). Therefore, we utilized them to differentiate the incidence of petroleum-related products in the environment. The maximum detected hopanes were at the C_{30} and/or C_{29} homologues, with the thermodynamically stable 17α(H),21β(H) configuration and minor 17β(H),21α(H)-hopanes (Table 1 and SM1). These isomeric configurations normally take place in crude oils and mature sedimentary rocks due to the diagenetic interconversion of the 17β(H),21β(H)-hopane precursors of bacterial origins (Peters & Moldowan, 1993). The hopane distribution ranged from C_{27} to C_{35} for the α,β-series with typically mature C-22 R/S (sinister/rectus enantiomers at carbon 22) pairs of the predominant homologs > C_{30} (Simoneit, 1984; Simoneit et al. 1990; Peters & Moldowan, 1993). High concentration distributions of the 22S hopane relative to the equivalent 22R isomer have been generally related to petroleum and vehicle engine exhaust (Simoneit, 1984, 1985). The 22S/(S + R) ratios for the extended hopanes of C31 and C32 varied from 0.29 to 0.8 in the sediments of the lagoon. They were 0.56 ± 0.11, 0.65 ± 0.06, and 0.62 ± 0.05 for sediments from IZ, MZ, and OZ, respectively. These ratios were comparable to the values reported for petroleum hydrocarbons and mature crude oil (Kvenvolden et al., 1990; Rushdi & Simoneit, 2002a, 2002b). Consequently, these ratios verified that the sources of the hopane compound series in the lagoon sediments were petroleum-related inputs.

The detection of steranes in the environment is generally related to petroleum emissions and or releases of lubricants from vehicular engines (Abas & Simoneit, 1996). Refinery activities, ship-washing discharges, and municipal wastewaters are also possible sources of these biomarkers (Laws, 1993). Hence, the sterane biomarkers are similarly valuable supporting tracers for oil-product contamination in coastal and urban environments (e.g., Aboul-Kassim & Simoneit, 1996; Albaiges & Cuberes, 1980; Rushdi et al., 2017a, 2017b, 2022a). The steranes in the sediments of Obhur lagoon ranged from C_{27} to C_{29} with primarily the 5α,14β,17β-series and minor 5α,14α,17α configurations and both occurring as the 20S and 20R epimers. The epimerization ratio of steranes at C-20, S/(S + R) for these samples, ranged from 0.10 to 0.91 for C_{27} and from 0.14 to 0.87 for C_{29}. They were 0.48 ± 0.11 in IZ sediments, 0.42 ± 0.12 in MZ, 0.46 ± 0.10 in OZ sediments for C_{27}, and 0.65 ± 0.15 in IZ, 0.0.62 ± 0.28 in MZ, and 0.66 ± 0.22 in OZ for C_{29} (Table 1). These ratios revealed that the sources of these biomarkers were petroleum residues (Peters & Moldowan, 1993; Simoneit, 1984).

Phthalates

The phthalates in the sediment samples of the lagoon were major compounds, including diisobutyl- dibutyl and dioctyl- phthalates. Their total concentrations ranged from 3.7 ng/g to 201.2 ng/g, with average concentrations of 37.4 ± 34.5 ng/g in the IZ, 58.6 ± 40.0 ng/g in the MZ, and 65 ± 68 ng/g in the OZ sediments (Table 1, and SM1, Fig. 3e). Dioctyl phthalate was the predominant plasticizer detected in the lagoon sediments. These man-made plasticizer compounds were not detected in the solvents, analytical blanks, and sample containers, indicating that they were the main components of the lagoon sediments. The total concentrations of these phthalates in the lagoon sediments were much lower than the levels measured in Campeche-Mexico (18292–21702 ng/g; Ramirez et al., 2019), Asalouyeh harbor coast of Iran (mean = 5180 ng/g; Arfaeinia et al., 2019), Arabian/Persian Gulf of Saudi Arabia coast (31–2799 ng/g; Rushdi et al., 2022a), similar to values in Santos Bay-Brazil (0.0–182 ng/g; Cesar et al., 2014), and relatively higher than the levels in the coast of Qatar (7.8 ± 0.7 ng/g; Rushdi et al., 2017a, 2017b).

Plastic waste and litter in marine ecosystems have been recognized as a serious environmental concern (Andrady, 2011; Eriksen et al., 2013; Fan et al., 2023; Isobe & Iwasaki, 2022; Jang et al., 2023; Peng et al., 2020; Perkins, 2015; Sivadas et al., 2022; Vegter et al., 2014). About 269,000 tons of plastic objects have been predicted floating on oceanic surface waters (Xanthos & Walker, 2017), which represent 60–95% of marine debris (Derraik, 2002; Surhoff & Scholz-Bottcher, 2016; Walker et al., 1997, 2006). Plasticizers, mainly phthalates, make up the mass chemical composition of plastics and are very stable compounds in the environment (Dong et al., 2019; Zhang et al., 2021). They are hazardous and harmful substances to marine life (Squillante et al., 2023; Tranganida et al., 2023).

Unresolved complex mixture (UCM)

Commonly, the GC traces of TEOM of environmental samples show the unresolved complex mixture (UCM) of branched and cyclic compounds above the baseline with the superimposing of the resolved compounds (Frysinger et al., 2003; Killops & Al-Juboori, 1990; Simoneit, 1984, 1985). The concentrations of UCM in the sediment samples of the lagoon were relatively high, ranging from ~ 0.0 to 4571.4 ng/g (Table SM1) with high concentration ranges in the three zones (Figs. 2f and 3f). They ranged from 27 to 2755 ng/g (average = 821 ± 1119 ng/g) in IZ, 0.0–4571.4 ng/g (average = 1041 ± 1539 ng/g) in MZ, and from 21 to 4433 ng/g (average = 1297 ± 1684 ng/g) in OZ (Table 1). These concentrations were lower than the values reported in sediments from the Arabian/Persian Gulf coasts of United Arab Emirates (10000–670000 ng/g; Tolosa et al., 2005), Iraq (71753–92540 ng/g; Rushdi et al., 2014a, 2014b), Saudi Arabia (14–11388 ng/g, Rushdi et al., 2022a), and relatively similar to Qatar (38–609 ng/g; Rushdi et al., 2017a, 2017b).

Fossil fuel utilization and oil spills are the main sources of the UCM. Generally, the refining process of crude oil produces diesel and gasoline, with the former having a broader UCM and the latter having a narrower UCM (Frysinger et al., 2003; Gough & Rowland, 1990; Simoneit, 1984, 1985). A TEOM of a sample with only biogenic sources such as terrestrial plants has no UCM (Simoneit & Mazurek, 1982). Thus, the UCM-to-resolved compound ratio (U:R ratio, Simoneit & Mazurek, 1982) is employed to evaluate the level of contamination from the input of petroleum-related products and oil spills. A U:R ratio value of > 1.0 suggests contamination by biodegraded petroleum-related products in sediments. The U:R ratios varied from 0.01 to 3.5 (Table SM1) and the average values were 1.00 ± 0.99 for IZ, 0.86 ± 1.17 MZ, and 1.23 ± 1.33 for OZ (Table 1). The estimated values of the U:R of the sediment samples of the lagoon indicated that the lagoon was comparatively contaminated with petroleum residues.

The ratio of UCM/n-Alkanes has been used to confirm and distinguish the sources of petroleum hydrocarbons in the environment, where the values > 2.0, > 4.0, and > 10 indicate petroleum, lubricating oil, and degraded petroleum, respectively (Harji et al., 2008; Tolosa et al., 2004). The ratio values ranged from 0.1 to 32.2 for the sediment samples of the lagoon. The averages were 6.8 ± 6.6 in IZ, 8.5 ± 7.6 in MZ, and 10.8 ± 10.9 in OZ. We have applied the ratios of UCM/n-Alkanes versus $\mathrm{CPI}_{(o/e)}$ to validate and differentiate the origins of hydrocarbons in the sediments of the lagoon (Fig. 5). Obviously, the main sources of these hydrocarbons in the lagoon were petroleum hydrocarbons.

The percentages of petroleum-related compounds, including petroleum n-alkanes, hopanes, steranes, and UCM, were relatively high ranging from 1 to 97% of the TEOM of the lagoon sediment. They were from 39.5 to 83.8% (average = 62 ± 15%) in IZ, 7.7–97.2% (average = 43 ± 33%) in MZ, and 18.8% to 90.2% (average = 54 ± 28%) in OZ. The fractions of phthalates from plastics varied from 0.70 to 5.6% in the sediments and from 1.8 to 4.7% (average = 2.9 ± 1.2%) in IZ, 0.7–4.7% (average = 3.2 ± 1.1%) in MZ, and from 1.3 to 6.3% (average = 4.0 ± 1.6%) in OZ (Fig. 5). The plasticizer fractions were similar to the values reported from the Gulf of Suez (3.3 ± 2.0%, Rushdi et al., 2009) and higher than in the Qatar coast (0.42 ± 0.72%, Rushdi et al., 2017a, 2017b).

Environmental effects

Coastal lagoons are important sources of organic and inorganic nutrients to marine environments (Por, 2012) with estimated primary production similar to upwelling areas (Knoppers et al., 1991). They are naturally very important and ultimate ecosystems for aquaculture projects but, are still, extremely influenced by anthropogenic inputs and developmental activities (Kjerfve, 1994). Urbanization and human activities have influenced the well-being conditions of the lagoons of the Red Sea coasts (Albarakati & Ahmad, 2019; Basaham et al., 2019). They affect key habitats, such as mangroves, coral reefs, seagrass stands, and bottom water fish nursing areas of the lagoons (Alamri et al., 2021; Aljahdali et al., 2021).

To evaluate the potential warning effects of these sediment hydrocarbons and phthalates, we've utilized the sediment quality assessment guidelines (SQAGs) approach developed by Long and Morgan (1990). This method involves testing various

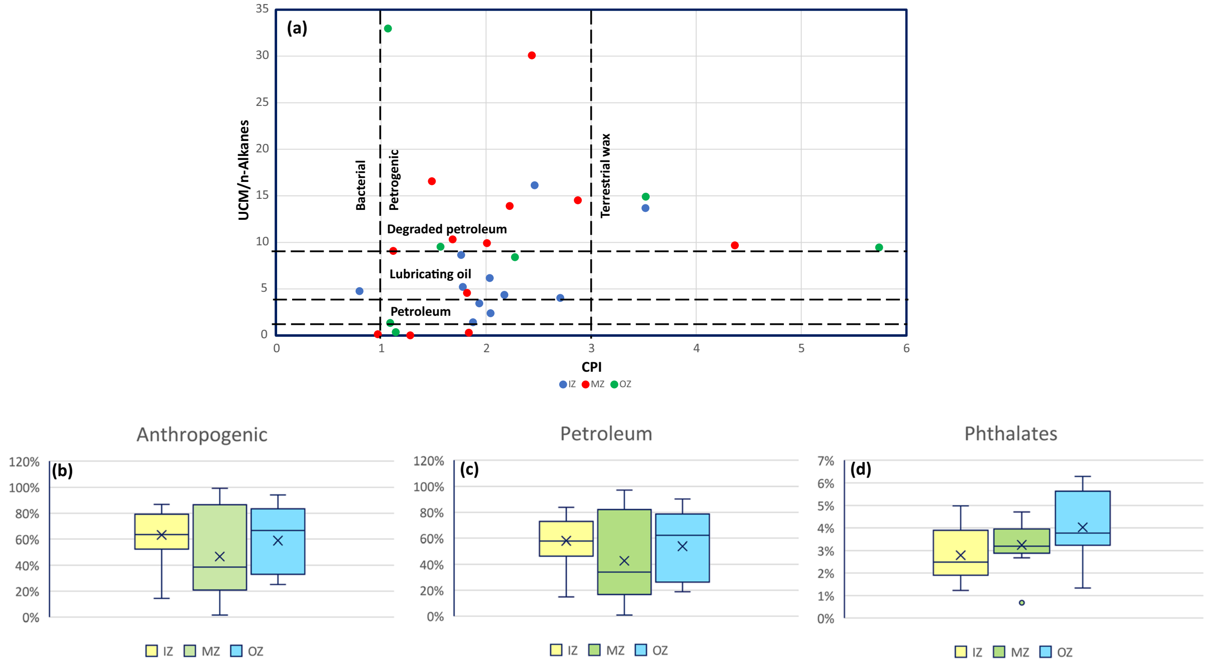

Fig. 5 Plots showing: **a** UCM/n-alkanes versus CPI parameters characterizing the different potential petroleum sources, **b–d** the percentages of anthropogenic, petroleum, and phthalates inputs in the three different zones of the lagoon

sediment contaminant concentrations against two ranges: the threshold effects level (TEL), which is the minimum level that could cause an effect, and the probable effects level (PEL), which is the lower limit range of contaminant concentrations that could cause damage to the biological system. Although n-alkanes have been reported to be non-toxic (Nilsen et al., 1988), there are no guidelines available for their regulation. Thus, we have used phthalates to assess the sediment quality of the lagoon. The TEL and PEL values for phthalate esters are 182 µg/kg and 2647 µg/kg, respectively, as reported by Long and Morgan (1990) and Long et al. (1995). For DBP, based on the characteristics of toxicology and environmental chemistry, Van Wezel et al. (2000) established environmental risk limits (ERLs), which was set at 0.7 mg/kg for DBP. The acceptable exposure threshold (AETs) for DNBP is 71 1400 µg/Kg, according to PTI (1988) and Bellar et al. (1986). The TEL for DBP is 2200 ng/g and the PEL is 17000 ng/g, as determined by Kingsley and Witthayawirasak (2020). Accordingly, based on the works mentioned above, the levels of DBP and total phthalates found in the sediments of the lagoon were below the TEL values. This indicates that the measured concentrations of phthalates should not have any significant harmful effects.

Increase in petroleum-related products and plasticizer wastes from many sources such as oil transfer docks, oil refineries, municipal sewage treatment plants, and public littering is anticipated to have impacts on the various habitats and associated species groups in the lagoons (Dicks, 1987; Loya, 1975; Rinkevich & Loya, 1979). The most sensitive early life stages of biota such as egg, larva, and young genera will be disturbed by these toxic chemicals. Other marine species, especially benthic species that depend on the lagoon for reproduction, growth, and protection, are harshly impacted. Thus, the presence, characteristics, distributions, and sources of crude oil contamination and other man-made chemical pollutants in the Obhur lagoon need further studies. These studies will facilitate the control and preventive measures to be undertaken to avert or at least reduce the inputs of these pollutants into the lagoons.

Conclusion

The TEOM hydrocarbons in the sediments of Sharm Obhur were mainly from petroleum residues (43 ± 33–62 ± 15% of the TEOM) with fewer inputs from vascular higher plants (23.4 ± 29.0–31.2 ± 34.4% of TEOM). The sources of petroleum hydrocarbons were largely from oil-related operations, spills, boat engines, oil transfer docks, oil refineries, and untreated water discharges into the lagoon. Other sources of pollutants in the sediments of the lagoon were phthalates from plastic littering. These petroleum residues and plasticizers will in due course have severe effects on the ecosystems of the lagoon and the adjoining Red Sea coastal environments.

Further studies are necessary to monitor and investigate the occurrence, characteristics, and distribution of organic and inorganic pollutants in the Red Sea coastal lagoons and their impacts on the key habitats of the area. These research and monitoring activities are critical to protecting the important ecosystems of the lagoons.

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Declarations

Conflict of interest The authors declare no competing interests.

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