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Photo-oxidation products of petroleum hydrocarbons in the Eastern Red Sea coastal waters

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Abstract

The level of dissolved oil residues as well as the oxygenated petroleum hydrocarbons in the seawater of the affected area of Jeddah coast, Red Sea was estimated. The dissolved/dispersed petroleum hydrocarbons concentration was found minimal. The minimum level may be attributed to the action of the weathering processes.

Selected samples of the dichloromethane extracts of seawater were fractionated, using silica gel columns, into four fractions eluted with solvents of increasing polarity. The fluorescence intensity of these fractions was measured at three different pairs of excitation and emission wavelengths using ultraviolet fluorescence spectrofluorophotometry (UVF). The total fluorescence intensity of the fractions at 280 nm excitation, 327 nm emission wavelengths and 310 nm excitation, 360 nm emission wavelengths exceeds the fluorescence intensity of the whole extract by more than two orders of magnitude. The estimation of higher molecular mass oxygenated compounds in the fourth fractions, read at 380 nm excitation, 430 nm emission wavelengths, indicates rather low concentrations of $<1 \mu\text{g l}^{-1}$ hydroxyppyrene equivalents. However, the polar fractions (eluted with acetone) account for more than 25% of the total fluorescence intensity of all fractions combined at each pair of wavelengths. The characterization of the products causing the fluorescence particularly the high molecular mass compounds still presents difficulties.

Preliminary GC/MS results revealed some evidence for petroleum hydrocarbon oxidation products. Alkyl isobenzofuranones have been found in some seawater extracts. This is an ongoing project to characterize and identify additional compounds from these extracts.

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1. Introduction

The coastal marine environment of the Saudi Red Sea is subject to direct and indirect influences of industrial facilities found along the Saudi Red Sea coast (i.e. Jeddah, Rabigh and Yanbu; MEPA, 1992). Oil refineries are considered as a major industrialized area along the Red Sea coast that may contribute most of the contaminants to the marine environment. Crude oil and oil products released into seawater undergo physical, chemical and biological transformations (i.e. evaporation, dissolution, adsorption, microbial degradation and photochemical oxidation). The composition of spilled oil in seawater is different from that of the original oil as a result of

weathering processes and is further changed with the passage of time by microbial decomposition and photochemical oxidation. Estimations of oil pollution in the marine environments are mainly based on measurements of polycyclic aromatic hydrocarbons (PAHs). Most of the PAHs are scarcely water soluble and potentially toxic to aquatic organisms. Recently, oxidation products (O-PAH) of petroleum hydrocarbons, results of photochemical transformation processes, have been suggested to be included in the monitoring of organic pollutants in the marine environment (Ehrhardt and Burns, 1993; Burns, 1993). It has been observed that photochemical oxidation proceeds in the presence of sensitizers (e.g. flavin, anthraquinone, etc.) and, if the parent hydrocarbons absorb solar UV radiation, in their absence. Photooxidation was also found to be favoured in nutrient-depleted seawater, and under intense solar radiation (Ehrhardt et al., 1992). Nutrients concentrations in the Red Sea are

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found to be low particularly in the central region (Weikert, 1987). Ahmad and Sultan (1989) reported that the annual mean based on the monthly average of the recorded short wave radiations along the eastern side of the Red Sea between 18°N and 23°N latitudes are 215 W m^{-2} . Photochemical oxidation products have been examined experimentally by several workers using crude oils, fuel oil and also individual PAHs under simulated and natural solar radiation (e.g. Ali et al., 1995; Burwood and Speers, 1974; Ehrhardt et al., 1992, 1997; Larson et al., 1977). They have concluded that the major products were peroxides, phenols, acids, carbonyls and γ -lactones. Moreover, many authors have detected O-PAHs in marine environments. For examples, some oxygenated aromatic hydrocarbons have been detected in seawaters from the upper part of Arabian Gulf (Ehrhardt and Douabul, 1984) and in Bermuda coastal waters (Ehrhardt and Burns, 1990). They found that the oxidation products were higher in quantity than the parent hydrocarbons. Burns (1993) has also estimated oxygenated petroleum hydrocarbons in marine organisms. Generally, the resulting oxidation products of hydrocarbons are more water-soluble than the parent compounds. Some of them have been shown to be quite toxic to aquatic organisms (Larson et al., 1977), while other products resemble known carcinogens. The estimation of oxygenated products using UVF is a sensitive technique (Burns, 1993), despite the fact that the oxidation products have lower fluorescence quantum yields than the respective parent hydrocarbons. Thus, Burns's (1993) technique was adopted in this study

with some modifications in the solvent system (see Section 2).

The available information concerning the concentrations of petroleum hydrocarbons in the marine environment of the Saudi Red Sea coast is still limited (see Al-Lihaibi and Al-Ghamdi, 1997). It appears that, no single study has been reported on the estimation of photochemical oxidation products of petroleum hydrocarbons in this area of the subtropical waters (i.e. Red Sea). Thus, this study was desirable to estimate the level of oil contamination as results of the 1996 fuel oil spill on the coastal area in the vicinity of Jeddah and also to verify the presence of oxygenated petroleum hydrocarbons.

2. Materials and methods

The study area (vicinity of Jeddah) is located in the central part of the eastern Red Sea coast (Fig. 1). This area is characterized by the presence of the Jeddah oil refinery. Therefore, the main sources of oil pollution in the study area are the refinery and the activities related to oil transportation (e.g. normal operational losses, accidental spill, etc.). Recently, the area was affected by a spill of bunker C fuel oil from the cargo ship "Liverpool-Bay". The accident was reported in March 1996. It was estimated that more than 200 tons of bunker C fuel were spilled. This type of fuel is equivalent to the characteristic of heavy crude oil and/or No. 6 fuel. The countermeasure of such type of oil spill on a marine environment

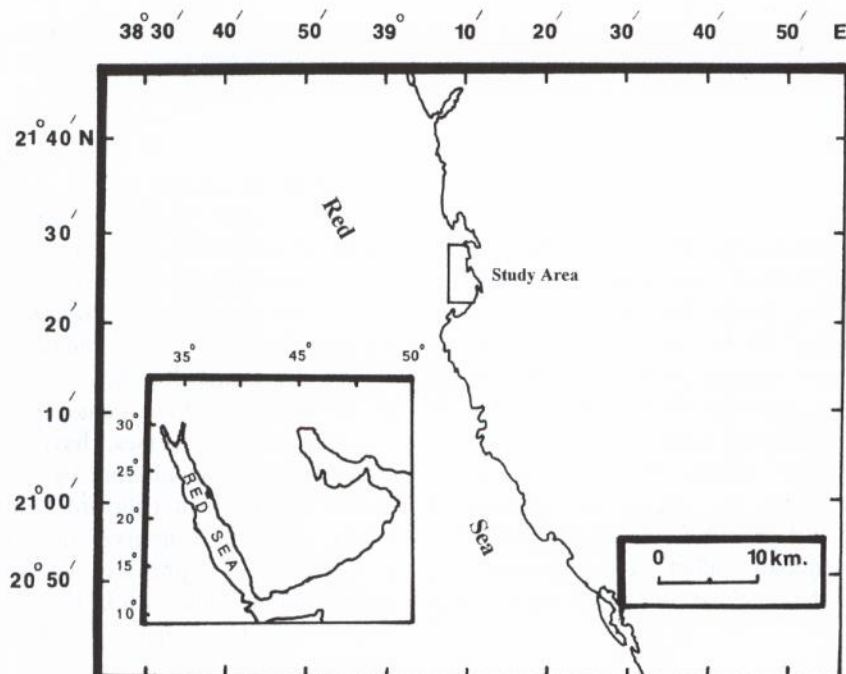


Fig. 1. Location of the sampling from the Eastern Red Sea (Jeddah Coast).

can be summarized as follows: (i) little or no evaporation, (ii) heavy contamination of intertidal area, (iii) weathering processes are very slow (iv) shoreline clean up is difficult under all condition. The study area was surveyed and found that the upper tidal area, coastal area and intertidal area were relatively affected with tarballs and tar mats (Fig. 2). The origin of the tars is attributed to the recent spill on the site (Liverpool Bay cargo ship). However, all the samples were collected from the subtidal areas and the depths of the area are variable.

The samples for this study were collected after completion of the clean-up operations, which was carried out under the supervision of the Presidency of Meteorology and Environment (formerly Meteorological and Environmental Protection Administration). Surface seawater samples were collected 18 months after the spill. Pre-cleaned amber glass bottles (ca. 2.8 l) were used for collection of

seawaters samples; they were closed with Teflon stopper before deployment. The collected samples (1 m depth) were preserved by adding aliquots of organic solvent (dichloromethane), shaken, and then transported to the laboratory for extraction and immediate analysis.

2.1. Extraction and analysis

All glassware was soaked in a solution of Decon-90 in distilled water (ca. 5%), then rinsed with tap water, distilled water, re-distilled water and finally baked in an oven (200 °C, 24 h). The organic solvents used were HPLC grade and were re-distilled through an all-glass still, using fractionated distillation. Silica gel adsorbent (60–120 mesh; BDH Chemical) was pre-extracted with dichloromethane (CH₂Cl₂) in a Soxhlet extractor and dried in an oven at 120 °C. The dried material was

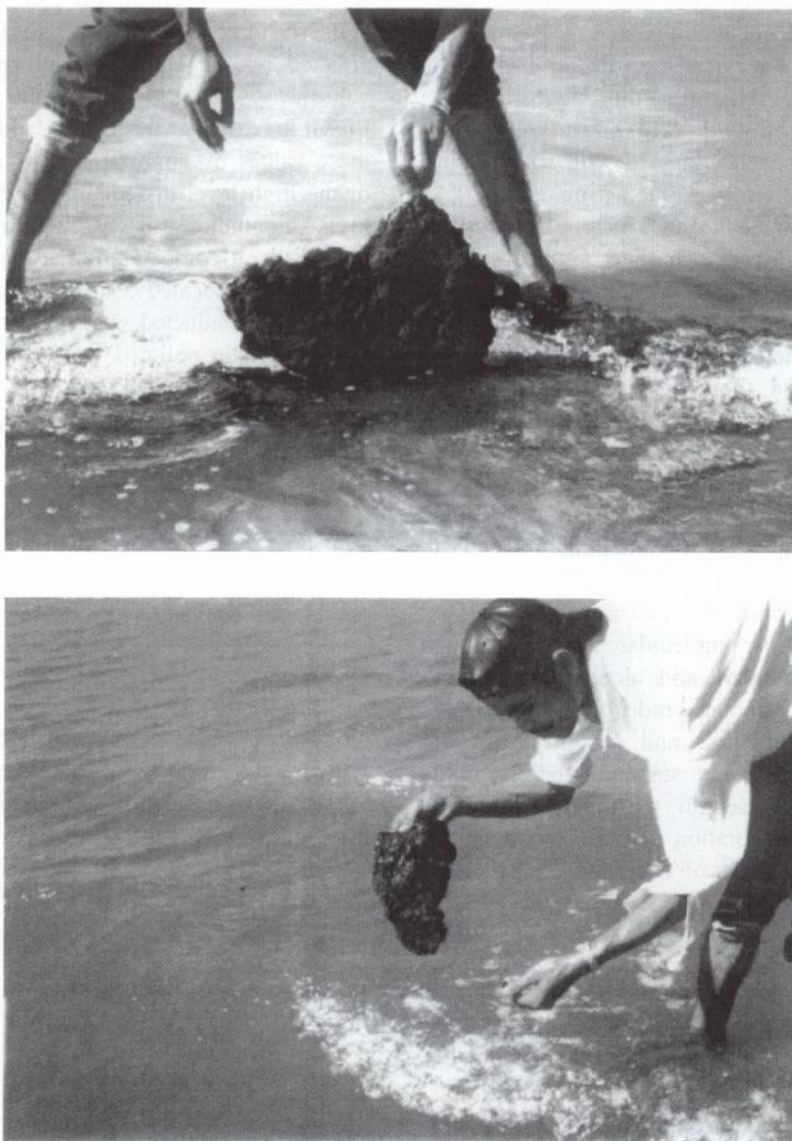


Fig. 2. Photographs by Al-Lihaibi taken in 1997 showing the tar mats in the intertidal zone of Eastern Red Sea (Jeddah coast).

deactivated with 5% re-distilled water and stored in an amber glass bottle. Anhydrous sodium sulphate was also pre-extracted with CH_2Cl_2 in a Soxhlet apparatus and was dried in an oven.

2.2. Petroleum hydrocarbons and oxidation products

Dissolved/dispersed petroleum hydrocarbons and their oxidation products were extracted from seawater with dichloromethane; the dried extractable organic matter (EOM) was weighed and re-dissolved in *n*-hexane. The dissolving quantity of the *n*-hexane (ca. 100 ml) was sufficient enough to avoid the quenching during measurement. Petroleum hydrocarbons were quantified by the intensity of their fluorescence (310 nm excitation, 360 nm emission wavelengths) using an ultraviolet fluorescence (UVF) Spectrofluorophotometer (Shimadzu RF-5000). The procedural blank was determined as water extracts and the value ($0.4 \mu\text{g l}^{-1}$ light Arabian crude oil equivalents) was subtracted from the sample concentrations.

Selected seawater extracts were purified and fractionated by column chromatography (column dimensions ca. 10×350 mm, each packed with 8 g silica gel). Four fractions (ca. 50 ml each) were obtained with solvents of increasing polarity of (i.e. *n*-hexane, *n*-hexane+10% dichloromethane, dichloromethane and acetone). The solvents of these fractions were evaporated, the residues were weighed on a micro electro-balance, were then re-dissolved in *n*-hexane, and each fraction was analyzed by UVF Spectrofluorophotometer. The estimation by UVF of aromatic hydrocarbons and their oxygenated derivatives has been adopted from Burns (1993). Briefly, three different pairs of excitation (exc.) and emission (em.) wavelengths (i.e. 280 nm exc./327 nm em., 310 nm exc./360 nm em. and 380 nm exc./430 nm em.) were applied to estimate the different classes of compounds. The last pair of wavelengths was used to estimate the oxygenated petroleum hydrocarbons, while the 280 nm exc./327 nm em. pair estimated the low molecular mass aromatic hydrocarbons and their quinone and alcohol derivatives (see Burns, 1993). Light Arabian crude oil was used as reference standard for the first and second pairs of wavelengths, and hydroxypyrene standard for the 380 nm exc./430 nm em. wavelength pair. The standards were used to construct calibration curves and also to qualitatively estimate the classes of compounds present in the four fractions.

GC/MS analyses of the aliphatic fractions were made on a Shimadzu GC-17A gas chromatograph equipped with a split/splitless injector, and a fused silica column ($25 \text{ m} \times 0.3 \text{ mm i.d.}$, $0.17 \mu\text{m}$ DB-5% phenyl/95% methyl-silicone) using helium as carrier gas. The GC conditions were as follows: temperature programming $40\text{--}300 \text{ }^\circ\text{C}$ at $5 \text{ }^\circ\text{C}/\text{min}$ then isothermal for 5 min; the injection port temperature was $250 \text{ }^\circ\text{C}$, and the helium flow rate ca. 2 ml min^{-1} . The end of the GC column

was introduced into the electron impact (EI) ion source of a Shimadzu QP-5000 quadrupole mass spectrometer. Typical mass spectrometer operating conditions were as follows: transfer line $230 \text{ }^\circ\text{C}$, ion source temperature $250 \text{ }^\circ\text{C}$, electron energy 70 eV. All samples were analyzed in full data acquisition (SCAN) mode between 50 and 500 Daltons per unit charge at 1 cycle/s.

3. Results and discussion

3.1. Extractable organic matter

The extractable organic matter (EOM) concentrations in surface seawater samples from the study area ranged from 0.8 to 6.1 mg l^{-1} (Table 1). It is noteworthy that the highest concentration of EOM was observed at Station 14. The average concentrations of EOM in seawater near the Jeddah oil refinery (Stations 11, 12, 13 and 14) were 5.3 mg l^{-1} . In the zone that was most affected by the Bunker C oil spill (Stations 1–10), the value was 3.3 mg l^{-1} . Thus, the average concentration of EOM in seawater near the Jeddah oil refinery exceeds these values by approximately 2 mg l^{-1} . The explanation of the high level in Jeddah refinery is obvious and is due to the continuous discharge from the refinery. To judge this result, a comparison was made with that study done on an area allocated 5 km southward from this area. The study was conducted on evaluating the organic pollutants from domestic polluted water samples. It has been found that the EOM ranged from 1.1 to 12.5 mg l^{-1} with an average of 3.35 mg l^{-1} (El-Sayed and Niaz, 1999).

Table 1

Concentrations of extractable organic matter (EOM) and dissolved and dispersed petroleum hydrocarbons (DDPHs) from the Eastern Red Sea water (Jeddah coast)

Station no.	EOM, mg l^{-1}	DDPHs, L.A.O. eq. $\mu\text{g l}^{-1}$ exc. 310/em. 360
1	3.4	1.3
2	3.2	1.3
3	5.6	18.0
4	5.8	10.8
5	2.0	2.7
6	n.d.	0.9
7	1.1	2.1
8	4.2	1.0
9	4.1	3.2
10	0.8	2.9
11	5.2	1.1
12	n.d.	2.0
13	4.5	17.2
14	6.1	1.4
Average	3.8	4.7

L.A.O. = light Arabian oil; eq. = equivalent; exc. = excitation; em. = emission; n.d. = not determined.

3.2. Petroleum hydrocarbons

Concentrations of dissolved/dispersed petroleum hydrocarbons (DDPHs) in seawater from the study area are also presented in Table 1. The concentrations ranged between 0.9 and 18.0 $\mu\text{g l}^{-1}$ with an average of 4.7 $\mu\text{g l}^{-1}$. It is noteworthy that Stations 3, 4 and 13 have high petroleum hydrocarbon concentrations and consequently these locations are considered polluted. However, most of the values found in this study are similar to those reported from surface seawaters of the Red Sea (Al-Lihaibi and Al-Ghamdi, 1997), and the Arabian Gulf (El-Samra et al., 1986; Badawy et al., 1993; Ehrhardt and Burns, 1993). The latter reference found that the DDPHs concentration in seawater samples from Saudi Arabian Gulf Coastal area following the 1991 major oil spill did not exceed 3.5 $\mu\text{g l}^{-1}$. Furthermore, the concentrations in the studied area are also comparable to those found in a variety of locations worldwide (e.g. Sen-Gupta et al., 1980; Fileman and Law, 1988). On the other hand, Awad (1984) reported higher concentrations of dissolved/dispersed petroleum hydrocarbons in seawater from the Jeddah area (50–250 $\mu\text{g l}^{-1}$) than those found in this study. The difference between values obtained in the present study and those of Awad (1984) may result from differences in the sampling locations. The study of Awad (1984) was mainly conducted beyond the barrier reef area (within the main navigation channel), which is subject to discharges of ballast water.

Keizer and Gordon (1973) have considered that the concentration of 2 $\mu\text{g l}^{-1}$ of petroleum hydrocarbons is the threshold between oil polluted and unpolluted waters. Remarkably, most of the seawater samples (64%) from the study area had concentrations below 2 $\mu\text{g l}^{-1}$. It is known that the influence of the oil refinery on the area obvious and also that the dissolution from batches of the tar balls and/or tar mats deposited on affected intertidal area is another source of input into the study area (Fig. 2). However, the plausible explanation for low concentrations of oil residues in seawater of this area is the effects of weathering processes (i.e. evaporation and photooxidation).

3.3. Oxygenated petroleum hydrocarbons

Estimation of aromatic hydrocarbons and the oxygenated derivatives in the individual fractions of water extracts are presented in Table 2. For the quantification, coloured fractions were diluted to colourless to avoid fluorescence quenching by biogenic material. The Stations 5, 7, 9 and 14 had the highest concentrations in nearly all fractions and to some extent at the three wavelengths pairs. Moreover, the total fluorescence intensities of the four fractions at 280/327 nm wavelengths and 310/360 nm exceed the signal fluorescence of the whole extract, which was read at 310/360 nm, by orders of magnitude. The concentrations of the fractions read at 280/327 nm were higher than those of the same fractions read at

Table 2

Fluorescence based concentrations ($\mu\text{g/l}$) and percentages (%) in each silica gel fraction of seawater extracts from the Eastern Red Sea (Jeddah coast)

Sample/ fraction	Light Arabian oil eq. exc. 280/em. 327 nm		Light Arabian oil eq. exc. 310/em. 360 nm		Hydroxy pyrene eq. exc. 380/em. 430 nm	
	Conc. $\mu\text{g l}^{-1}$	%	Conc. $\mu\text{g l}^{-1}$	%	Conc. $\mu\text{g l}^{-1}$	%
1						
F1	23.8	21.3	15.6	31.0	0.08	26.6
F2	21.7	18.4	8.0	15.9	0.08	26.6
Subtotal	45.5	39.7	23.6	46.9	0.16	53.2
F3	34.2	30.6	14.1	28.0	0.08	26.6
F4	31.9	28.5	12.6	25.0	0.06	20.0
Subtotal	66.1	59.1	26.7	53.0	0.14	46.6
2						
F1	26.9	34.0	14.7	39.7	0.07	17.5
F2	14.1	17.8	5.5	14.8	0.06	15.0
Subtotal	41.0	51.8	20.2	54.5	0.13	32.5
F3	26.53	33.5	11.7	31.6	0.16	40.0
F4	11.56	14.6	5.1	13.7	0.11	27.5
Subtotal	38.09	48.1	16.8	45.3	0.27	67.5
3						
F1	13.1	27.4	7.1	35.5	0.08	25.8
F2	7.13	14.9	3.1	15.5	0.06	19.3
Subtotal	20.23	42.3	10.2	51.0	0.14	45.1
F3	17.3	36.2	6.5	32.5	0.10	32.2
F4	10.2	21.3	3.3	16.5	0.07	22.5
Subtotal	27.5	57.5	9.8	49.0	0.17	54.7
4						
F1	25.6	25.9	10.7	23.6	0.05	13.1
F2	34.8	35.2	20.0	44.2	0.07	18.4
Subtotal	60.4	61.1	30.7	67.8	0.12	31.5
F3	19.2	19.4	7.0	15.4	0.20	52.6
F4	19.2	19.4	7.5	15.5	0.06	15.7
Subtotal	38.4	38.8	14.5	30.9	0.26	68.3
5						
F1	128.0	24.4	17.50	23.3	0.07	25.9
F2	120.0	25.0	21.53	28.7	0.07	25.9
Subtotal	248.0	49.4	39.03	52.0	0.14	51.8
F3	122.0	25.4	18.8	25.0	0.07	25.9
F4	109.0	22.7	17.1	22.8	0.06	22.2
Subtotal	231.0	48.1	35.9	47.8	0.13	48.1
7						
F1	89.0	42.0	20.4	23.8	0.09	28.1
F2	39.0	18.0	19.3	22.5	0.08	25.0
Subtotal	128.0	60.0	39.7	46.3	0.17	53.1
F3	52.0	25.0	30.6	35.2	0.06	18.7
F4	31.0	15.0	15.4	17.9	0.09	28.1
Subtotal	83.0	40.0	46.0	3.1	0.15	46.8
9						
F1	150.0	26.3	7.1	22.3	0.08	25.8
F2	140.0	24.5	8.5	26.8	0.08	25.8
Subtotal	290.0	50.8	15.6	49.1	0.16	51.6
F3	141.0	24.7	6.3	19.8	0.08	25.8
F4	139.0	24.3	9.8	30.9	0.07	22.5
Subtotal	280.0	49.0	16.1	50.7	0.15	48.3
14						
F1	110.00	24.4	10.31	21.0	0.11	30.5
F2	95.00	21.1	10.64	21.6	0.08	22.2
Subtotal	205.00	45.5	20.95	42.6	0.19	52.7
F3	122.00	27.10	14.08	28.7	0.06	16.6
F4	123.00	27.30	14.02	28.5	0.11	30.5
Subtotal	245.00	54.40	28.1	57.2	0.17	47.1

eq. = equivalent; exc. = excitation; em. = emission.

310/360 nm. It is interesting to note that a similar pattern was observed in the study of Burns (1993). In the latter study, the non-polar fractions (1, 2 and 3) exhibited high concentrations compared with the same fractions at 310/360 nm. Burns (1993) stated that the fluorescence of the low molecular weight aromatic hydrocarbons (1–3 ringed) and their oxygenated derivatives was most intense at emission 327 nm when excited at 280 nm. Therefore, the high concentrations of the four fractions of the seawater extracts at 280/327 nm could be attributed to the presence of these compounds. In contrast, the concentrations in the individual fraction at 430 nm emission wavelength (hydroxypyrene equivalents) were $<1 \mu\text{g l}^{-1}$. These results also are in good agreement with those reported by Burns (1993). The percentage of the fluorescence intensity of the polar fractions (acetone) was more than 25% of the total intensity of the four fractions at each wavelength pair. Ehrhardt and Burns (1993) have estimated the fluorescence intensity in four fractions of seawater extracts from the Arabian Gulf following the 1991 oil

spill. They believed that the rapid degradation of dissolved oil residue in the coastal waters of the Arabian Gulf is due to the interactions of photo-oxidation and microbial decomposition. The present investigations also lead us to the conclusion, that the photooxidation process plays key factor for the decline of the oil pollution in seawater. While, the evaporation and the microbial degradation processes are slow in action due to the characteristic of this fuel type and the scarcity of nutrients.

The preliminarily GC/MS results of the seawater extracts show some evidence for the presence of low molecular mass oxygenated hydrocarbons. For example, homologous alkyl acetophenones or isobenzofuranones have been tentatively identified. Fig. 3B shows a mass spectrum of isobenzofuranone compound (?) extracted from the seawater of the Saudi Red Sea Coast. The peaks at m/z 65, 77, 91 and 119 indicated that the backbone of the compound is definitely monocyclic aromatic. The absence of the peak at m/z 43 (CH_3CO) may rule out the hypothesis that the compound is alkylacetophenone. Ehrhardt and Burns (1993) have reported C_3 and C_4

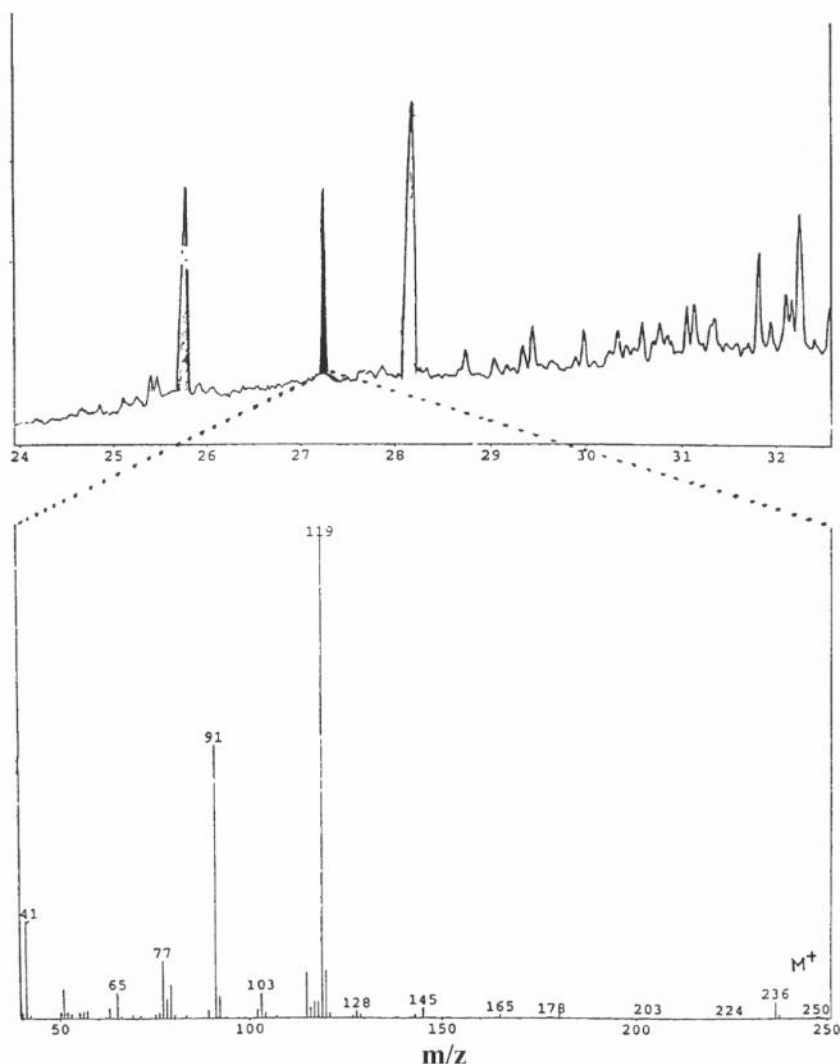


Fig. 3. Mass spectrum (background corrected) of alkyl isobenzofuranone compound from dichloromethane fraction (Fraction 3) of seawater extract from the Eastern Red Sea (Jeddah coast).

alkyl acetophenones in seawater extract from the Arabian Gulf, and afterwards they re-identified these compounds as dimethylisobenzofuranone. Ehrhardt et al. (1997) identified alkylisobenzofuranones as photooxidation products of alkyl-naphthalenes.

4. Conclusions

Despite oil-related activities, concentrations of dissolved/dispersed petroleum hydrocarbons in Saudi Red Sea coastal water (study area) were low and within the range of results reported previously. The effect of degradation processes (chemical and microbial) on oil residues in the Saudi Red Seawater is remarkable and to a large extent responsible for the decline of oil residue concentrations in seawater. The discharge of effluents from the oil refinery is reflected in relatively elevated concentrations of petroleum hydrocarbons in adjacent seawater.

On the basis of the fluorescence intensities of the total extracts and individual fractions of selected seawater samples, the sum of the aromatic and oxygenated hydrocarbons in these fractions exceeds the concentration of the whole extracts by orders of magnitude. The percentage of the fluorescence intensity of the polar fractions (acetone) of most extracts represents more than 25% of the total intensity of the four fractions. Contributions of biogenic materials to the fluorescence of individual fractions of the extracts can not be neglected, but these have been minimized. The presence of alkyl acetophenones or isobenzofuranones in the polar fraction proves the hypothesis of the presence of oxygenated hydrocarbons in the Saudi Red Sea environment. However, a full characterization of these compounds (e.g. high molecular weight oxygenated aromatic hydrocarbons) is still looking for a reliable method since the UVF quantification underestimates the dissolved oil residue concentration due to the presence of many oxidation products which lower the fluorescence quantum. Moreover, the toxicity of the oxidation products of polycyclic aromatic hydrocarbons requires careful investigations.

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