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## Baseline

## Distribution and sources of polycyclic aromatic hydrocarbons in the surface sediments of Gorgan Bay, Caspian Sea

Peyman Eghtesadi Araghi\*, Kazem Darvish Bastami, Shirin Rahmanpoor

Iranian National Institute for Oceanography and Atmospheric Science (INIOAS), No. 3, Etemadzadeh St., Fatemi Ave., Tehran 1411813389, Islamic Republic of Iran

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## ABSTRACT

The level and source of 16 polycyclic aromatic hydrocarbons (PAHs) were investigated in the surface sediments from the Gorgan Bay, Caspian Sea. The sum of 16 PAHs ( $\Sigma$ PAH16) concentrations varied from 107.87 to 516.18 ng g<sup>-1</sup> dry weight, with average value of 270.96 ± 150.47 ng g<sup>-1</sup> dry weight. Ecological risk assessment of PAHs, indicated that adverse biological effects caused by acenaphthene, naphthalene, fluoranthene and Pyrene occasionally and frequently may take place in the sediments of Gorgan Bay. PAHs source identification demonstrated that the PAHs come from mixed and pyrogenic origin.

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Polycyclic aromatic hydrocarbons (PAHs) are among the most important contaminants in the marine environment which are produced from different resources. PAHs are a large group of organic compounds with two or more fused aromatic rings. They have mutagenic and carcinogenic activity that are usually present in the air, water and aquatic system, soils and sediments (Neff, 1979).

These compounds are generally generated by natural and anthropogenic processes and can be introduced into the environments through various routes. Anthropogenic input from incomplete combustion, oil spills, urban runoff, domestic and industrial wastewater discharges, as well as atmospheric fallout of vehicle exhaust and industrial stack emission have caused significant accumulation of these compounds in the environments (Nadal et al. 2004; Durand et al. 2004).

The Gorgan Bay located at the southeastern section of the Caspian Sea with 60 km length and 12 km width, is a semi-enclosed basin, as it receives no wave's energy from the Caspian Sea. The Bay is mostly affected by the currents originating from inside the basin. This area is best known for its high economical and ecological importance as a fishing and recreational ground because of appropriate biological conditions for aquatic animals. The Gorgan Bay is the ultimate sink for water flows from Gharehso River. The Gorgan River faces the Caspian Sea at the vicinity of the Bay mouth at the northeast part (Bastami et al., 2012).

Little information is available on PAH in the sediment of Gorgan Bay, therefore; the objective of this study was to determine the concentration and possible sources of PAHs in the sediment of Gorgan Bay.

Fifteen stations were selected for this study and sediments of sites were collected by using van veen grab in August, 2010 (Fig. 1). The samples are collected in aluminium container and kept it in the freezer (−20 °C).

Sediment samples obtained from one location was mixed and a composite sample was prepared. Then, these sediment samples (200 g of sediment) were freeze dried (freeze-drier Model: OPR-FDB-5503, Korea) at −40 °C for 24 h to a constant weight. 10 g of each sample were mixed with 250 ml of *n*-hexane and dichloromethane mixture (1:1 ratio). The mixture was placed for 8 h in Soxhlet. The combined extracts were evaporated on a rotary vacuum evaporator until 15 ml was left. To eliminate sulphur and their compounds, 3 g of active copper was added to the mixture and allowed to pass through a folded filter paper (Whatman GF/C, 24micron) for 24 h. The mixture was concentrated in a rotary (Buchi B-490) to a volume of 5 ml. Samples were allowed to pass through a column containing 10 g silica gel in *n*-hexane, 1–2 g anhydrous sodium sulphate and 10 g alumina. Then, 30 ml of *n*-hexane and dichloromethane mixture (9:1 ratio) were added to the column. Samples were concentrated again in a rotary evaporator to a volume of 5 ml. The concentrated extract was dried under nitrogen steam (N-E VAP 112, USA). The residue was dissolved in 1 ml of acetonitrile (MOOPAM, 1999).

The PAHs were detected with a Hewlett–Packard 1100 HPLC equipped with an Agilent-1100 fluorescence and UV detector. Injection volume was 20 µl. The initial mobile phase was 60% acetonitrile and 40% HPLC water for 8 min, which was then gradiently changed to 100% acetonitrile in 5 min, held at 100% for 22 min, then decreased to initial phase. In this analysis the column type was RESTEK, Pinnacle II PAH (150 × 3.2 mm, 5 µm) and temperature was 25 °C.

\* Corresponding author. Tel.: +98 9124755635.

E-mail address: [peghtesadi@gmail.com](mailto:peghtesadi@gmail.com) (P.E. Araghi).

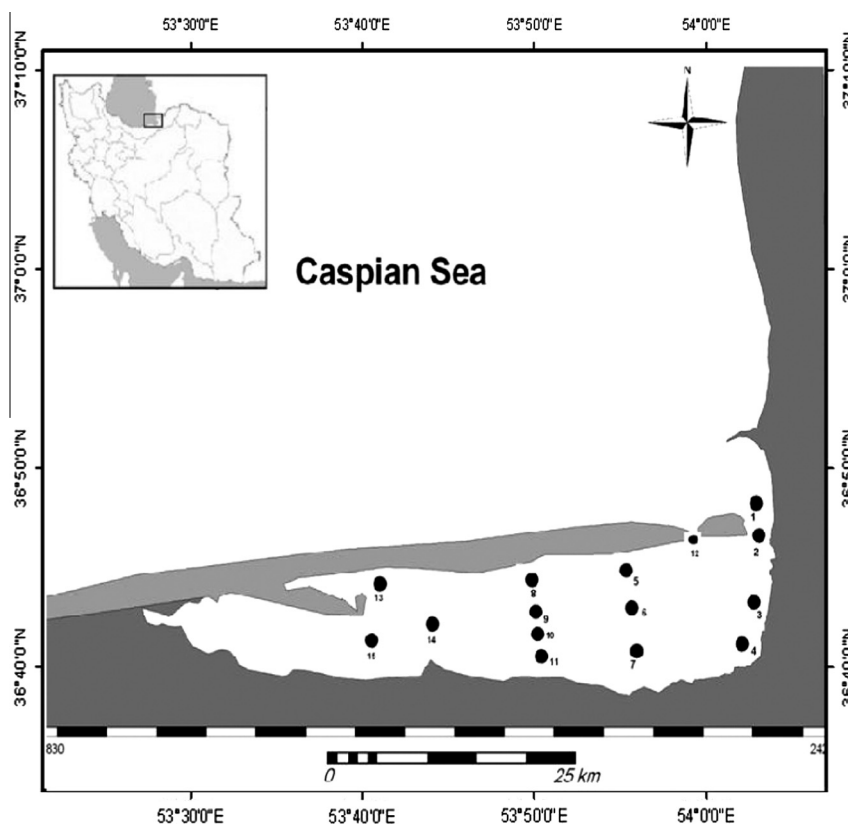


Fig. 1. The locations of the sampling sites at the Gorgan Bay.

The sixteen most toxic PAHs listed by the USEPA (Bashe and Baker, 1990) were assessed in three sample media. The abbreviations used for the identified PAHs are: naphthalene, N; acenaphthylene, AC; acenaphthene, ACE; fluorene, F; phenanthrene, Ph; anthracene, A; fluoranthene, Fl; pyrene, Py; benzo (a) anthracene, B(a)A; chrysene, Ch; benzo (b) fluoranthene, B(b)Fl; benzo (k) fluoranthene, B(k)Fl; benzo (a) pyrene, B(a)Py; indeno (1, 2, 3-c, d) pyrene, IPy; dibenz (a, h) anthracene, DB(ah)A; benzo (ghi) perylene, B(ghi)Pe.

Replicate samples, certified reference materials IAEA-417 and procedural blanks were used as quality control procedures. All the obtained values for PAHs in CRM were in the reported range. Reproducibility and recovery were high (85–110%) with relative standard deviation (RSD) 4–11%. To measure the quality control, the procedural blanks were periodically analysed for each batch of 10 samples. Quantitative analysis was conducted on a three-point linear calibration of PAH solution, obtained by dilution of the certified standard mixture of 16-PAH (kit 610-N-Supelco4-7351). The method detection limits (MDLs) of PAHs were determined with a signal-to-noise ratio (S/N) of 3. The MDLs were between 0.1 and 1 ng/g. Satisfactory linearity was obtained with values of the correlation coefficient R above 0.99.

For determination of organic matter, sediment samples were dried at 70 °C for 24 h and then combusted in an oven at 550 °C for 4 h. Total organic matter, as described by Abrantes et al. (1999), was measured by the following equation:

$$\text{Total organic matter (TOM, \%)} = (B - C/B) \times 100$$

where B and C are the weights of dried sediment before and after combusting in the oven, respectively. Sediment grain size analysis was made from sediment samples collected in separate polythene bags. For that, samples were air dried and sieved through a mechanical sieve to remove shells, debris, etc. Dried sediment samples were

subjected to size fraction analysis following the procedure of Wentworth (1992). Hundred grams of sample was taken and sieved through a 62  $\mu\text{m}$  mesh-sized screen for 10 min in a mechanical sieve shaker. The sample that remained in the sieve was weighed and treated as sand. The sediment samples which passed through the sieve were the silt and clay. The silt and clay fractions were then separated by means of pipette method, described by Lindholm (1987).

Regression Correlation was employed for a better understanding of the relationship between the concentration of PAHs, Clay and TOM by using statistical package of SPSS 19.

The sand, silt and clay contents averaged  $23.38 \pm 27.56\%$ ,  $36.61 \pm 18.3\%$ , and  $39.76 \pm 14.40\%$ , respectively (Table 1). Gorgan Bay depth ranges from 0.62 to 4.12 m with an average value of  $1.84 \pm 1.14$  m. The highest and the lowest depths were observed at stations 6 and 10, respectively. Regarding to the analysis, TOM content in sediment of the Gorgan Bay was 1.75–9% (averaged  $4.45 \pm 1.72\%$ ) which exhibited the highest and the lowest amounts at stations 15 and 14, respectively. As proposed by Marin et al. (2008), ecological quality status is classified into three levels according to organic matter content: high-good (less than 5%), moderate (between 5% and 10%) and poor-bad (greater than 10%). As illustrated in Table 1, TOM content of all stations was less than the presented above range (1.75–9%) in the current study. According to their proposal, stations 1, 2, 3, 4, 7, 8, 9, 10, 12, 13, and 14 showed a high-good quality while the remaining stations (5, 6, 11 and 15) should be classified as moderate.

Ten PAHs were measured at detectable levels in the sediment from Gorgan Bay. The highest concentration of individual PAH was Fluoranthene, while B (a) anthracene showed the lowest concentration in the sediment of Gorgan Bay (Fig. 2). The concentration of  $\Sigma$ PAHs ranges  $107.87$ – $516.18$   $\text{ng g}^{-1}$  dry weight with an average value of  $274.31 \pm 152.29$   $\text{ng g}^{-1}$  dry weight (Table 2).

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