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Polyaromatic Hydrocarbons (PAHs) and Metal Evaluation After a Diesel Spill in Oaxaca, Mexico

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Abstract Pollution in the marine environment due to a diesel spill takes days to months to complete natural remediation owing to its low volatility. Metal and PAH contamination caused by an accidental diesel spill were studied. V, Ni and Hg levels increased immediately after the spill, while PAH levels decreased after 1 month (79.4–7.6 μg kg^{-1}). At the diesel spill point, fluoranthene exceeded acute and chronic levels, although most of the PAHs were within the range of low effects. In fish body burden, the highest bioaccumulation factor (2.63 for naphthalene) was related to the lower molecular weight PAHs.

Keywords Diesel spill · PAH body burden · TEL · PEL

Fuels transportation in Mexico from refineries to distribution bulk plants is mainly performed by pipeline. At the Pacific side of Mexico, in Salina Cruz, Oaxaca there is one of the main distribution centers of refined products that control pipelines. This place has suffered many uncontrolled illegal or accidental pipeline flow outs. Effects in aquatic biota at the area are not well documented and restoration plans must be based on data concerning to the type and concentrations of pollutants. On April, 2002 a diesel spill was detected on a 16" pipeline from Minatitlán, Veracruz to Salina Cruz. Diesel spilled out from the pipeline to San Pedro stream, Xadani estuary and the Superior Lagoon mouth (Fig. 1). These all aquatic systems are used mainly for local fisheries.

Based on the data reported by Cronk et al. (1990), direct deaths of adult fish occur infrequently, and the diesel spill itself had a very minor, localized and short-term impact on the Antarctic marine environment (Cripps and Shears 1997). Longer-term effects of spills on biota are less obvious and follow-up studies of individual events. However, it is not clear whether sufficient studies can be conducted to discriminate the effects of single events, especially in areas of repeated spills (Lytle and Perckarsky 2001). It is in these cases when the sediment analysis becomes important because it keeps historical spill records (Washington Department of Ecology 2003). To evaluate the spatial and temporal effects due to the diesel spill, sediments (metals and polyaromatic hydrocarbons, PAHs) and dwelling native fish (body burden PAH) were collected and evaluated.

Materials and Methods

According to the configuration of the aquatic systems and the location of the hot spot, 22 sampling points were located as follows (Fig. 1). San Pedro stream: five sampling points. Xadani estuary: seven points distributed from San Pedro stream to the Superior Lagoon mouth. Piedra estuary: three sampling points, considering as a reference site. Superior lagoon: seven sampling points. A Van Veen grab of 20 kg capacity was used to collect sediment samples in duplicate. Fish samples were also taken in duplicate in both climatic seasons: dry (May) and rainy season (September).

Metals (Pb, Cd, Cu, Cr, Ni, Zn, V, As and Hg) in sediments were measured by flame atomic absorption

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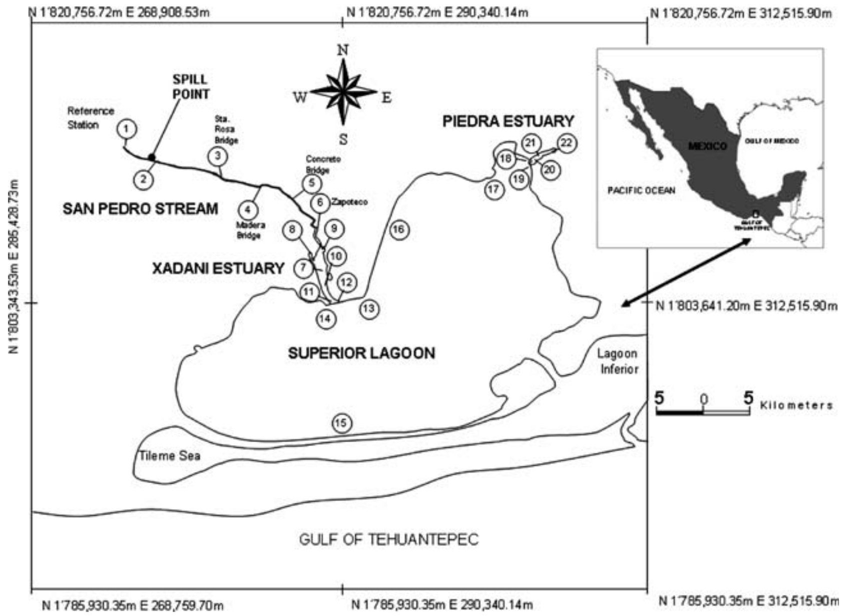

Fig. 1 Sampling points (in number) at Salina Cruz, Oaxaca, Mexico

spectrometry (Perkin Elmer 2800), using for digestion EPA 3050A and for analysis EPA 7000 methods (US EPA 1998; DL = 0.01 μg g^{-1}). A total of 22 PAHs (including 16 EPA priority PAHs) were analyzed using EPA 3550B for sediment extraction, EPA 3630C for purification, and EPA 8310 methods for detection and quantification (DL = 2 μg kg^{-1}). The PAHs fish tissues were extracted according to EPA 3540 method. Lipid content was measured additionally using the method described by Kelet and Lederer (1974). Fortified recoveries of sediment samples varied between 42 and 75%, and tissue samples between 28 and 62%. All PAH recoveries met quality control limits.

Results and Discussion

Table 1 shows different metals in sediments represented by median values calculated from concentrations measured in each station of the aquatic system, and the sediment quality guidelines reported in Macdonald et al. (1996). Total concentrations of each metal in the study area increased during rainy season, which is related with erosive processes of washing out from the drainage basin and the carry out of ions due to effects of the climatic season. Metal concentrations that were superior during dry season in which the spill occurred were V, Ni and Hg, the first two indicators of diesel.

Although samples were taken four months apart (May–September), and therefore the data may be is not comparable between seasons, the objectives were (a) to compare the immediate effects of the diesel spill with the effects after the diesel recovery, and (b) to establish, from numerous individual studies, an association between the concentration of each chemical measured in the sediment and any Adverse Biological Effect (ABE) observed per season. The lower value, referred to the threshold effect level (TEL), represents the concentration below which adverse biological effects are expected to occur rarely. The upper value, referred to as the probable effect level (PEL), defines the level above which adverse effects are expected to occur frequently (Table 1). By calculating TELs and PELs, three ranges of chemical concentrations are consistently defined: (1) the minimal effect range within which adverse effects rarely occur, (2) the possible effect range within which adverse effect occasionally, and (3) the probable effect range within which adverse biological effects frequently (Long et al. 1995).

At the area, ABE are expected to occur rarely caused by Cd and Ni, Pb on rainy season and Hg on dry season. At the whole area, ABE are expected to occur rarely caused by Cd and Ni, Pb in rainy season and Hg in dry season. Cr during rainy season at the Xadani estuary and San Pedro stream, as well as Ni during dry season showed higher levels than the threshold levels, therefore may be caused adverse effects frequently.

Figure 2 shows the total PAH pattern in sediment (values represent the sum of concentrations measured in the sampling point). Results showed a decrease in the total PAH levels, being lower after 4 months of the spill, which may be due to rains, sediment removals and washed outs.

The concentration of PAHs in marine and estuary sediments varies widely (Table 2). Areas that receives industrial waste water showed levels of 100 mg kg^{-1} or higher. At areas without anthropogenic activities, PAHs values ranges from μg kg^{-1} (Kennish 1997). According to the PAHs levels, the most impacted ecosystem by the

Table 1 Metals (mg kg^{-1}) contents in sediments (dry weight)

Site	S	Pb	Cd	Cu	Cr	Ni	Zn	V	As	Hg
SPR	D	9.9	2.66*^{a}	19.7*	39.5	46.7*$^{a''}$	68	<5.0	8.3*^{a}	0.6*^{a}
SPR	R	17.1	1.75*^{a}	12.7	26.3	24.4*^{a}	51	<5.0	2.7	0.09
SPS	D	18.0	2.66*^{a}	16.6	29.2	13.8	44	110.5	7.4*	0.36*^{a}
SPS	R	35.1*	2.96*^{a}	23.6*	157.9*^{a}	29.2*^{a}	81	123.0	7.9*	0.05
PE	D	23.5	2.65*^{a}	21.6*	60.1*	26.9*^{a}	45	95.4	3.4	0.19*^{a}
PE	R	38.3*	2.92*^{a}	28*	102.7*^{a}	31.3*^{a}	61	148.9	4.7	0.07
XE	D	26.7	2.82*^{a}	19.8*	49.5	31.5*^{a}	59	113.3	8.4*^{a}	0.33*^{a}
XE	R	38.0*	3.39*^{a}	18.5	165.2*$^{a''}$	32.6*^{a}	69	107.7	6.5	0.04
SL	D	20.4	2.69*^{a}	11.0	34.5	16.8*	29	<5.0	4.6	0.15*
SL	R	36.7*	2.16*^{a}	8.9	28.6	20.3*	27	<5.0	3.4	0.04
TIA	D	20.8	2.74*^{a}	16.6	39.9	25.7*^{a}	42	112.8	6.9	0.26*^{a}
TIA	R	37.4*	2.96*^{a}	18.4	164.6*$^{a''}$	31.2*^{a}	66	108.3	6.5	0.04
TEL*		30.2	0.68	18.7	52.3	15.9	124	NA	7.2	0.13
ERL^{a}		46.7	1.2	34	81	20.9	150	NA	8.2	0.15
PEL″		112	4.21	108	160	42.8	271	NA	41.6	0.7
ERM ~		218	9.6	270	370	51.6	410	NA	70	0.71
DL		<5	<1	<5	<5	0.3	<25	<5	<0.4	<0.02

S Season, *D* dry, *R* rain, *SPR* San Pedro reference, *SPS* San Pedro site, *PE* Piedra estuary, *XE* Xadani estuary, *SL* Superior Lagoon, *TIA* Total impacted area, *TEL* Threshold Effect Level, *ERL* Effects Range Low; *PEL* Probable effect level, *ERM* Effects range median, *DL* detection limit. Symbols mean that values exceed the criteria

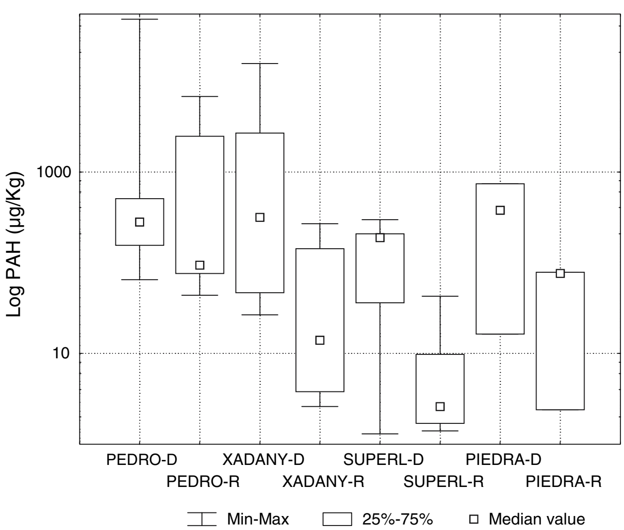

Fig. 2 Total PAHs in sediment. *D* dry season, *R* rainy season

diesel spill was San Pedro stream, followed by Xadani estuary due to the diesel migration. In the Superior Lagoon and Piedra estuary, there were not detected high levels of PAHs. However, at the reference site, there were detected 22 different PAHs, some of them lower-molecular-weight PAH compounds, but in a similar pattern.

The most impacted sites immediately after the spill were located at the hot spot and at the sampling point number 11 (Fig. 1). The highest total PAHs level was 48.1 mg kg^{-1}, at the spill point (San Pedro stream). On the rainy season, the most impacted site was Santa Rosa Bridge (sampling point number 3), where the hydrodynamic was slow and let the accumulation of fine sediment particles adsorbing the organic compounds. Considering 22 PAHs, acenaphthylene was the only one below the detection limit (Table 2) may be due to its high fugacity (Krein and Schorer 2000), and the relation input/decay. However, Piedra estuary, considering as a reference site, showed the presence of the 22 PAHs, but at lower levels than the rest of the sampling points. Between seasons (dry to rainy), a significant decrease in PAHs concentration was observed, mainly because of the rain that causes the contaminant dispersion.

Total concentrations of PAHs by themselves are not the best indicator of the potential toxic effects into the aquatic ecosystem biota. Therefore, the sediment quality criteria, which are based on effect evaluation of a single compound, can be used as a screening tool to evaluate the environmental quality of the sediment and to identify areas that potentially have undesirable effects. Table 3 shows sediment quality guidelines reported by Long et al. (1995), comparing with the incidence of probable effects.

According to the criteria described in Table 3 and considering the PAHs content of the whole area, most of the aquatic ecosystems are within the minimal effect range (88–95.5% are <ERL), 2–12% refers to the incidence in which effects may be presented occasionally and 0–4.5% is the incidence for probable effects. Anthracene, phenanthrene, fluoranthene, pyrene, benzo(a)anthracene and chrysene reached the thresholds able to induce chronic

Table 2 PAHs (μg kg^{-1}) in sediment. Values represent the sum of the sampling points

PAH	SP Re-D	SP Re-R	SPS Re-D	SPS Re-R	PE-D	PE-R	XE-D	XE-R	SL-D	SL-R
A	<3.6	<3.2	<4.0–2.40	<4.8–19	<2.5–3.8	<6.7	<1.25	<3.6	<2.6–1.3	<2.8–1.4
B	<3.6	<3.2	4.0–76	<4.8–200	<2.5–3.4	<6.7	<33.8–176	<3.6–6.9	<2.6–1.5	<2.8
C	<3.6	<3.2	<4.0–65	<4.8–240	<2.5–3.4	<6.7	<33.8–215	<3.6–9.1	<2.6–1.7	<2.8
D	<3.6	<3.2	<4.0–20	<4.8–15	<2.5	<6.7	<33.8–135	<3.6	<2.6	<2.8
E	<3.6	<3.2	<2.6–100	<4.8–530	<2.5–19	<2.8–28	<3.0–1540	<3.3–39	<2.6–10	<2.8
F	<3.6	<3.2	<5.48	<4.8	<4.8	<2.5	<1.25	<3.6	<2.6	<2.8
G	2.2	<3.2	<5.48–490	<4.8–80	<2.5–6.3	<6.7	<1.25	<3.6	<2.6–2.3	<2.8
H	<3.6	<3.2	1.4–110	<4.8–520	<2.5–45	<6.7	<3.0–3540	<3.6–63	<2.6–24	<2.8
I	<3.6	<3.2	1.4–380	<4.8–68	<2.5–10	<6.7	<3.0–362	<3.6–6.7	<2.6–3.4	<2.8
J	3.9	<3.2	2.3–520	<4.8–380	<2.5–59	<6.7	<3.0–4070	<3.6–48	<2.6–32	<2.8
K	42	<3.2	5–5500	<3.2–890	<2.5–70	<6.7	3.3–2270	<3.6–25	<2.6–34	<2.8–4.2
L	8.8	<3.2	<4.0–1200	<4.8–210	<2.5–12	<6.7	<14–429	<3.6	<2.6–4.4	<2.8
M	<3.6	<3.2	<2.6–520	<4.8–210	<4.8	<6.7	<3.0–2160	<3.6–54	<2.6–44	<2.8
N	110	1.8	2.9–9800	<3.6–44	<2.5–95	<2.8–9.2	<14–92.2	<3.6	<2.6–36	<2.8–5.9
O	84	1.6	5–7500	<3.6–390	<2.5–68	<2.8–8.8	<14–296	<3.6	<2.6–21	<2.8–5.8
P	34	<3.2	<4–3700	<4.8–680	<2.5–37	<6.7	<2.9–202	<3.6	<2.6–14	<2.8
Q	39	<3.2	<4–3000	<4.8–790	<2.5–44	<2.8–5.9	3.2–179	<3.3–17	<2.6–15	<2.8–5.0
R	25	1.8	4.2–3500	<4.8–570	1.6–30	<2.8–6.4	<1.25–15	<3.3–3.1	<2.6–23	<2.8–3.4
S	28	<3.2	<4–2200	<4.8–560	2.6–32	<6.7	<1.25–2.8	<3.6	<2.6	<2.8
T	19	<3.2	<4–1800	<4.8–450	1.9–20	<6.7	<1.25–5.2	<3.6–4.8	<2.6–5.1	<2.8–2.9
U	27	<3.2	<4–2400	<4.8–610	3.1–29	<6.7	<1.25–6.1	<3.3	<2.6–10	<2.8–3.0
V	39	89	<2.6–660	20–260	<2.5–100	<2.8–3.3	<1.25–3.8	<3.6–7.5	<2.6–3.4	<2.8–2.6
W	18	<3.2	<5.48–1900	<4.8–430	2.5–22	<6.7	<3.0–6.0	<3.3	<2.6–6.8	<2.8
X	8.4	<3.2	<4.0–940	<4.8–160	1.7–12	<6.7	<1.25–2.4	<3.6	<2.6–4.0	<2.8
Y	20	<3.2	<5.5–1500	<4.8–450	3.0–20	<6.7	<1.25–4.7	<3.3	3.2–7.0	<2.8–3.2

Re Reference, *D* dry season, *R* rainy season, *SP* San Pedro stream, *SPS* San Pedro site, *PE* Piedra estuary, *XE* Xadany estuary, *SL* Superior Lagoon, *A* Naphtalene, *B* 2–Metilnapthalene, *C* 1-Metilnaphtalene, *D* Biphenil, *E* 2,6 Dimetil-naphtalene, *F* Acenaphtylene, *G* Acenaphtene, *H* 2,3,5-Trimetilnaphtalene, *I* Fluorene, *J* Dibenzothiophene, *K* Phenanthrene, *L* Anthracene, *M* 1-Metilphenantrene, *N* Fluoranthene, *O* Pyrene, *P* Benzo(a)Anthracene, *Q* Chrysene, *R* Benzo(b)Fluoranthene, *S* Benzo(k)Fluoranthene, *T* Benzo(a)Pyrene, *U* Benzo(a)Pyrene, *V* Perylene, *W* Indene(1,2,3-cd)Pyrene, *X* Dibenzo(a,h)Anthracene, *Y* Benzo(g,h,i)Perylene, (<) detection limit

Table 3 Frequency of the effects (in percent) defined by the comparison between the criteria values and the sediment PAHs concentrations

PAH	ERL (mg kg^{-1})	ERM (mg kg^{-1})	SQC-Ch (mg kg^{-1})	SQC-Ac (mg kg^{-1})	<ERL	ERL-ERM	>ERM	>SQC-Ch	>SQC-Ac
A	240	1500	2400	14000	88.6	4.54	4.54	2.27	0
B	85.3	1100	190	–	88.6	4.54	2.27	4.54	–
C	600	5100	113	1494	93.2	0	2.27	2.27	2.27
D	665	2600	850	49500	95.5	2.27	0	2.27	0
E	261	1600	1600	55000	93.2	2.27	2.27	2.27	0
F	384	2800	1200	115000	93.2	2.27	2.27	2.27	0
G	430	1600	18000	450000	95.5	2.27	2.27	0	0
H	63.4	260	12000	–	95.5	2.27	2.27	0	0
T	4022	44792	–	–	88	12	0	–	–

A Phenanthrene, *B* Anthracene, *C* Fluoranthene, *D* Pyrene, *E* Benzo(a) anthracene, *F* Chrysene, *G* Benzo(a) pyrene, *H* Dibenzo(a,h)anthracene, *T* Total, *ERL* Effects range low, *ERM* Effects range median, *SQC-Ch* Sediment quality criteria-chronic, *SQC-Ac* Sediment quality criteria-acute

toxicity in 2.3–4.5% of the aquatic systems. The only compound that reached the threshold of acute toxicity was fluoranthene in 2.3% of the cases, represented by the sampling point located at the spill point during dry season.

Figure 3 shows the PAHs found in fish tissues. PAHs concentrations in aquatic organisms are highly variable. Reported values range from 0.01 to greater than 5,000 mg kg^{-1} dry weight for individual contaminants. Elevated

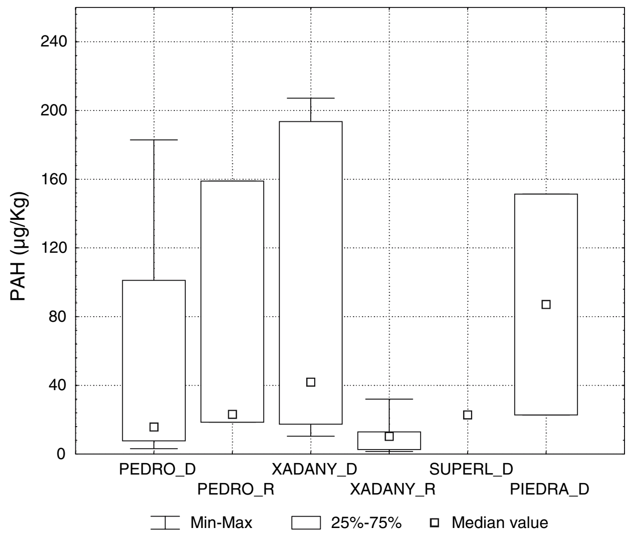

Fig. 3 Total bioaccumulated PAHs in fish tissue. *D* dry season, *R* rainy season

concentrations of PAHs in marine organisms often occur in areas receiving chronic hydrocarbons discharges (Tanabe 1994). At the study area, major concentrations were found in fish collecting in San Pedro stream, followed by Xadani estuary after 1 month of the diesel spill (dry season). Fish tissues from Superior Lagoon showed the lowest PAHs levels. The criteria values for human diet according to US EPA (2000) are as follows (ppb day^{-1}): acenaphthene (60), anthracene (300), fluoranthene (40), fluorene (40) and pyrene (30). Based on these values, fish tissue concentrations were lower than human daily diet.

The bioaccumulation factor indicates the potential of a compound to accumulate in a biological tissue. The accumulation depends on the compound lipophobicity, its bioavailability and the capacity of the organisms to metabolize it. These factors are calculated according to the level found in the tissue comparing with the concentration in the environment, either water or sediments. The mean bioaccumulation factors for the sampling points, obtained from the concentrations detected in sediments, are presented in Table 4. The bioaccumulation values for fish and sampling point ranges in San Pedro from 0.002 to 30.0, Xadani from 0.0012 to 8.5 and Lagoon from 0.15 to 1.4.

Lower-molecular-weight PAH compounds (naphthalene and methylnaphthalene) were mainly found in fish tissues. The preferential accumulation of these compounds is determined by their solubility and bioavailability, related to

Table 4 Mean bioaccumulation factors for PAHs in fish tissues

PAH	SP Re-D	SP Re-R	SPS-D	SPS-R	PE-D	PE-R	XE-D	XE-R	SL-D	SL-R
A	ND	ND	0.01	0.26	2.63	ND	ND	ND	1.46	ND
B	ND	ND	0.20	0.01	1.74	ND	0.18	0.93	0.93	ND
C	ND	ND	0.20	ND	0.97	ND	0.12	0.26	ND	ND
D	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
E	ND	ND	0.34	ND	0.10	ND	0.04	0.14	ND	ND
F	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
G	ND	ND	ND	0.07	0.19	ND	ND	ND	ND	ND
H	ND	ND	0.46	0.01	ND	ND	0.02	0.14	ND	ND
I	ND	ND	ND	0.03	0.66	ND	0.05	0.00	ND	ND
J	ND	ND	0.08	0.02	ND	ND	0.02	0.20	ND	ND
K	0.10	ND	0.01	0.01	0.42	ND	0.03	0.21	0.05	ND
L	0.22	ND	ND	0.01	0.92	ND	0.002	ND	0.20	ND
M	ND	ND	0.05	ND	ND	ND	0.002	ND	ND	ND
N	0.03	1.28	ND	0.11	0.25	ND	0.10	ND	0.04	ND
O	0.04	ND	ND	ND	0.23	ND	0.02	ND	0.04	ND
P	0.06	ND	ND	ND	0.31	ND	0.48	ND	0.07	ND
Q	0.04	ND	ND	ND	0.22	ND	0.03	0.00	0.04	ND
R	0.05	ND	ND	ND	0.33	ND	0.33	0.00	ND	ND
S	ND	ND	ND	ND	0.13	ND	1.46	ND	ND	ND
T	ND	ND	ND	ND	0.16	ND	0.42	0.00	ND	ND
U	ND	ND	ND	ND	0.20	ND	0.35	0.00	ND	ND
V	ND	ND	ND	ND	0.02	ND	0.00	0.00	ND	ND
W	ND	ND	ND	ND	0.15	ND	0.28	0.50	ND	ND
X	ND	ND	ND	ND	0.13	ND	0.79	ND	ND	ND
Y	ND	ND	ND	ND	0.13	ND	0.72	0.26	ND	ND

Re reference, *D* dry season, *R* rainy season, *ND* not determinated, *A* Naphtalene, *B* 2-Metilnaphtalene, *C* 1-Metilnaphtalene, *D* Biphenil, *E* 2,6 Dimetil-naphtalene, *F* Acenaphtylene, *G* Acenaphtene, *H* 2,3,5-Trimetilnaphtalene, *I* Fluorene, *J* Dibenzothiophene, *K* Phenanthrene, *L* Anthracene, *M* 1-Methilphenantrene, *N* Fluoranthene, *O* Pyrene, *P* Benzo(a)Anthracene, *Q* Chrysene, *R* Benzo(b)Fluoranthene, *S* Benzo(k)Fluoranthene, *T* Benzo(e)Pyrene, *U* Benzo(a)Pyrene, *V* Perylene, *W* Indene(1,2,3-cd)Pyrene, *X* Dibenzo(a,h)Anthracene, *Y* Benzo(g,h,i)Perylene the octanol-water partition coefficient, molecular weight, exposure route and incorporation of the PAHs which involves the habits of native biota (Conell and Miller 1984).

The PAH body burden differ widely among estuarine and marine organisms due to three principal factors: (1) variable concentration of PAHs in coastal environments from highly polluted systems to those that are pristine, (2) different degrees of bioavailability of the compounds, and (3) variable capacities of the organisms to metabolize them (Hellou et al. 1995). Because PAHs tend to accumulate in sediments, benthic organisms may be continuously exposed to the contaminants. However, sediment-sorbed PAHs have only limited bioavailability to marine organisms, which greatly reduces their potential toxicity.

Metal levels in sediments for which adverse biological effects may occur rarely or occasionally are Cd and Ni. Cr may cause adverse effects only during rains in the Xadani estuary and San Pedro stream. Ni during dry season showed higher levels than the threshold levels, therefore may be caused adverse effects frequently.

The behavior of PAHs showed a decrease of the total concentrations after 1 month of the spill, being the concentrations lower during the rainy season. The most impacted ecosystem was the San Pedro stream, followed by Xadani estuary due to the diesel migration. In the hot spot, fluoranthene overcame the acute and chronic approaches, although most of the PAHs levels are within the range of low effects.

The body burdens of PAHs in fish tissue did not overcome the approaches of human diet established by US EPA. In fish tissues, the highest accumulation were related to the lower-molecular-weight PAH compounds.

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